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16. ABSTRACT

This manual documents the Quality Assurance Program for Region V, U.S. EPA, that will produce a numerical estimate of the reliability of all data values reported or used by the Region. Revisions will be made per the requirements of the finalized Quality Assurance Plan of the Agency. The elements of a quality assurance program are discussed, including Region V's QA Policy Statement, Objectives and Milestones, Quality Assurance Management, Personnel, Facilities, Equipment and Services, Review of Program Plans, Project Plans or Study Plans, Data Collection, Data Processing, Corrective Actions, Data Quality Assessment, Data Quality Reports, Chain of Custody and Specific Guidance.

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QUALITY ASSURANCE PROGRAM
GUIDELINES AND SPECIFICATIONS
CRITERIA AND PROCEDURES
REGION V

U.S. ENVIRONMENTAL PROTECTION AGENCY
QUALITY ASSURANCE OFFICE
SURVEILLANCE AND ANALYSIS DIVISION
CHICAGO, ILLINOIS
JANUARY 15, 1980

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GLOSSARY

1. IDENTIFICATION OF OFFICE OR LABORATORY SUBMITTING QA PLAN

Document Title:	Quality Assurance Program Guidelines and Specifications Criteria and Procedures Region V	Ref. NO.: EPA-905/4-80-001
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Individual Responsible:	John McGuire Regional Administrator Region V U.S. Environmental Protection Agency 230 South Dearborn Street Chicago, Illinois 60604 (FTS)353-2000	
Individual Responsible for QA:	William H. Sanders III Director Surveillance and Analysis Division U.S. Environmental Protection Agency Region V 536 South Clark Street Chicago, Illinois 60605 (FTS)353-3808	
Plan Prepared By:	James H. Adams, Jr. Chief Quality Assurance Office Surveillance and Analysis Division U.S. Environmental Protection Agency Region V 536 South Clark Street Chicago, Illinois 60605 (FTS)353-9604	
Submission Date:	January 15, 1980	
Calendar Year Covered:	Interim document will be used pending finalization of Agency Quality Assurance Plan.	

Summary of environmental monitoring or measurement activities performed by Region V:

Quality assurance activities have been planned for 1980 in Air Quality Monitoring, Air Enforcement, Dredge and Fill, Ambient Water Quality Monitoring, Water Quality Enforcement, Public Water Supply Management and Great Lakes Monitoring. There are also special studies, contracts and other activities that require evaluation for quality assurance.

INTRODUCTION

Environmental Protection Agency (EPA) Policy, enunciated in memoranda of May 30 and June 14, 1979, requires participation in a centrally-managed Quality Assurance Program by all EPA Regional Offices, Program Offices, EPA Laboratories, and the States. This includes those monitoring and measurement efforts mandated or supported by EPA through regulations, grants, contracts, or other formalized agreements. The Quality Assurance Programs for the States in Region V will be cooperatively developed with them and implemented through the Regional Office.

The Office of Research and Development (ORD) has been given the responsibility for developing, coordinating, and directing the implementation of the Agency Quality Assurance Program. In addition, an Agency Quality Assurance Advisory Committee, chaired by ORD and with representatives from the Program Offices, Regional Offices, Staff Offices, and the States, has been established to coordinate this effort.

At this point, the distinction between two concepts -- quality assurance and quality control -- becomes relevant. "Quality Assurance" is defined here as an organization's total program for assuring the reliability of data it produces. A QA Plan is a document presenting the policies, objectives, management structure, and general procedures which comprise this total program. "Quality Control" refers to the detailed and specific procedures used to ensure the quality of data produced by a particular measurement activity. For example, a QA Plan for laboratory instruments would state that calibration needs to be addressed as an element of data collection activities. It would not, however, give instructions about how to do this calibration; these instructions represent quality control.

As an initial step in implementing this policy, Quality Assurance Plans (Programs) must be prepared by all EPA-supported or -required environmental monitoring and measurement activities per the specifications of EPA's guidance document MQA 001-79.

EPA policy is quite clear that the Agency Quality Assurance Program encompasses all environmentally related measurement activities undertaken by the Regional Offices, Program Offices, State Program Offices, and Laboratories; supported by these divisions through contracts, grants, or other formalized agreements; or required by them through regulations. A very broad definition of "environmentally related measurement activities" has been adopted. It includes all field and laboratory investigations which generate data. The measurement of chemical, physical or biological parameters in the environment; health and ecological effects studies; clinical and epidemiologic investigations;

studies involving laboratory measurements or simulated environmental events are covered under this definition and all such activities must be covered by a Quality Assurance Plan.

This document describes the Quality Assurance Program for Region V, U.S. EPA, that will produce a numerical estimate of the reliability of all data values reported or used by the Region.

2. QUALITY ASSURANCE POLICY STATEMENT, REGION V

It is the policy of EPA, Region V that there shall be sufficient quality assurance activities conducted within the Region to assure the collection of data which meet the requirements of the Environmental laws and regulations that require implementation by EPA in Region V.

The Regional Administrator has the overall responsibility for implemenation of the Agency's quality assurance program for valid data quality. The Director of the Surveillance and Analysis Division (S&A), through the Chief of the Quality Assurance Office (CQAO), assures that quality assurance objectives are met for each monitoring project conducted within Region V. This responsibility also includes external monitoring activities of States, local agencies, contractors and others covered by the Agency quality assurance plan.

The immediate objective of the Quality Assurance Office is to insure that the quality of data collected, reported or used by the Agency is properly documented and that the data are sufficiently accurate and precise to meet the Agency's quality assurance objectives.

The following activities shall be carried out in accordance with Agency mandates specified in document MQA 001-79, and existing Agency regulations.

The Quality Assurance Program will consist of:

1. An adequately trained staff for implementation of the Region's quality assurance program as approved by ORD.
2. Equipment procurement and maintenance shall meet specifications required by regulations, approved methodology, or appropriate EPA guidelines and shall be approved by the CQAO. These requirements shall appply to all Region V monitoring activities and to State and local agencies when Federal funds are expended.

3. Analytical methods and procedures for all monitoring programs shall conform to EPA approved methodology when applicable, and shall include quality control measures. All methods and procedures shall be documented "cookbook fashion" and reviewed and approved or revised as required by Agency regulations and guidelines. Their revisions and updates shall be by the appropriate Agency mechanism, based on recommendations from the CQAO.

4. The Regional Administrator, based upon recommendations from the CQAO, through the Director, Surveillance and Analysis Division, shall approve State and local agency Quality Assurance policies and programs.

5. Region V and State and local laboratory and field monitoring facilities shall perform system and performance audits. These facilities shall participate in interlaboratory audits managed by the AQAC and coordinated with EMSL-RTP, EMSL-Cincinnati and EMSL-Las Vegas.

6. Data processing shall be documented, reviewed and revised as required by the Region's Quality Assurance Program and approved by the Office of Research and Development. Quality control measures must assure accurate data from analysis by Region V, State and local agencies. Data shall be validated according to criteria which shall follow EPA guidelines and regulations.

7. Directors of the several divisions in Region V have responsibilities for the quality of data collected and used in the performance of tasks required. These responsibilities are corroborated under this policy. The CQAO will coordinate the implementation criteria for validation of required data.

8. Standard operating procedures for air monitoring activities in Region V, State and local agencies for site selection, audits, evaluations, maintenance and enforcement shall be developed, documented and reviewed per the requirements of 40 CFR Part 58.

9. The CQAO shall report continuously on all Quality Assurance programs to program managers.

CONCURRENCE James H. Adams Jr. Jan. 14, 1980
Chief, Quality Assurance Office, Date
Surveillance & Analysis Division,
Region V

CONCURRENCE William H. Sanders III 1/15/80
Director, Surveillance & Analysis Date
Division, Region V

APPROVED J C McGuire 1-20-80
Regional Administrator Date
Region V

3. OBJECTIVES AND MILESTONES

The primary goal of the Region V, quality assurance program is to define and improve the reliability (accuracy and precision) of data generated and used by the Region, per Headquarters' mandate and Agency regulations. There must be a mechanism for so doing. In order to measure or estimate changes in data quality, the quality must be expressed in measurable (numerical) terms. Therefore, the first priority in the Region V quality assurance program is to establish and implement a method to define and quantitate the program product - data quality. This includes data from Regional programs, State and local agencies, grants and contracts. Each program that collects data is to be quality assured by a comprehensive evaluation and review process such that all of the activities that influence the quality of data are performed by appropriated trained staff, by methods acceptable to EPA on instruments that are approved and maintained and each data collection activity has a documented quality controlled program.

MILESTONE 1: Interim Region V Quality Assurance Program will be developed by the QAO by January 15, 1980. This program will be amended and updated to meet the Agency's final QA requirements for 1981 within 90 days after final guidance from Headquarters becomes available.

MILESTONE 2: All Regional Program Offices that are engaged in a field sample collection activity shall prepare a field sampling and quality control manual which documents their methods of sample collection, preservation, field custody, field instrument calibrations and field quality control protocol, plus any other requirements specified in Section 8 of this document, by April 15, 1980. These documents will be submitted to the QAO for review and recommendations to the Regional Administrator for approval by Sampling programs

Included are air, hazardous waste, toxic substances, priority pollutants, public water supply, ambient surface and ground water, NPDES and Great Lakes. Programs are to be updated per the requirements of new Agency regulations or guidelines.

MILESTONE 3: All Laboratories in the Surveillance and Analysis Division engaged in analysis of samples shall document their methodology and quality assurance/control program per the specifications in Section 8 of this document, by April 15, 1980 and submit such documentation to the QAO for review and recommendations to the Regional Administrator for approval. Programs are to be updated per the requirements of new Agency regulations or guidelines.

MILESTONE 4: All State's Water Agency(s) shall document their field and laboratory methodology and quality assurance/conrol program per the specifications in Section 8 of this document according to the dates specified in each State's 106 grant condition by the Regional Administrator. These documents are to be forwarded to the respective State Coordinator for processing through the media manager and the S&A Division to the QAO for review and recommendations to the Regional Administrator for approval as required by Agency regulations.

MILESTONE 5: All State and Local Air Agency(s) shall document their field and laboratory methodology and quality assurance/control program per the specifications in Section 8 of this document by January 1, 1980 to the respective State Coordinator for processing through the respective media managers and the S&A Division to the QAO for review and recommendations to the Regional Administrator for approval.

OBJECTIVE: Manage the quality assurance functions in Region V that impacts all factors that influence data quality in the Region's FY 80 program plan. The factors to be considered are personnel, equipment, procurement, methodology, legal requirements, organizational responsibilities where QA policies must be carried out and other factors. The implementation of an effective program will insure objectivity, self review and documentation so that cost effectiveness in the program is assured. Objectives have been identified for each program decision unit for FY 80, which are depicted in Appendix 1.

MILESTONE 1: Key action steps(milestones) have been finalized with due dates for objectives listed under each decision unit for FY 80, which is also depicted in Appendix 1.

OBJECTIVE: To establish an interraction at all levels of management such that QA principles are implemented.

MILESTONE 1: These interractions are in place and are illustrated in Appendix 2.

OBJECTIVE: To have QA resources assigned to QAO in proportion to need, rather than programs controlling resources by which priorities in those programs preclude resource commitment to QA as the program planning process specifies and the National QA program mandates.

MILESTONE 1: During the planning process for FY 81, the QAO will identify all activities that have QA requirements. Assess resource needs for enumeration of those QA activities. Formulate FY 81 QAO Zero Based Budget activities with QA committments. This resource assessment will encompass implementation of the Region's FY 81 QA plan (program) as approved by ORD.

4. QUALITY ASSURANCE MANAGEMENT

4.1 Introduction

The current quality assurance program that is functional in Region V during FY 80 evolved from the program planning process and is carried out under restrictions which are placed on QA by resource commitments and priorities that are established by programs which provide those resources. The organizational structure of Region V which relates to data collectors and decision making based on results of collected data is shown in Appendix 3.

A description of the Organization for present QA related activities follows:

WATER DIVISION: Has responsibilities in the public water supply, ambient surface and ground water and wastewater programs. The administration of these programs through grants results in data collection by State and local personnel. Resources (Appendix 1) for quality assurance are provided through Decision Units B-224 (Ambient Water Quality Monitoring) and C-215 (Public Water Supply Management).

AIR AND HAZARDOUS MATERIALS DIVISION: Has responsibilities for air programs, hazard waste management, pesticide and toxic substances. Programs are managed through grants and contracts. Technical and field support is provided by the S&A Division through activities of the District Offices, Technical Support Branch and the Central Regional Laboratory. Resources for quality assurance are provided through Decision Units A-235 (Air Quality Monitoring), A-305 (Air Enforcement), and A-305 (Air Enforcement Unleaded Gas Inspections).

ENFORCEMENT DIVISION: Has responsibilities for enforcement action in the various programs for compliance with Agency regulations. QA of data collection is important to the validation of data so that it can be defended in legal processes. Resources are provided for QA in the A-305 Decision Unit for PSD monitoring. However, QA support is provided in the B-303 Decision Unit by the QAO without resources being provided by the Enforcement Division.

PLANNING AND MANAGEMENT DIVISION: Maintains data processing facilities and handle data for special studies and STORET. Although the data unit processes data collected by other organizations, it produces final reports from data which may require summary or collation for final data reporting. Thus, it is in the overall process, a data producer. QA has no resource support for this division. QA programs have not been employed.

S&A DIVISION: Is responsible for surveillance and analysis in the various water, air, waste and toxic substance programs. Technical support, monitoring and project studies are carried out for the program offices. Resources for QA in these various functions of the S&A Division are those described under the other divisions. S&A Division branches support QA programs by auditing, sample collection, and special studies.

GREAT LAKES NATIONAL PROGRAM OFFICE: The Great Lakes are monitored under this program through grants and contracts for sample collection and shore laboratory analysis, as well as, the operation of the ship for open waters and shipboard analysis by contract. Technical and field support is also provided by the S&A Division through activities of the District Offices, Technical Support Branch and the Central Regional Laboratory. Resources for QA is provided under Decision Unit B-241 (Great Lakes).

4.2 Quality Assurance Management Plan

In this context the implementation of a quality assurance program is deemed as a management endeavor which attempts to interface all activities which impact data quality, be they management, technology, statistics, monitoring or maintenance. In order to assure the data quality, each of the numerous activities must respond to the basic needs from which data becomes possible.

When one realizes that the simplest item may become a critical item in data collection, it then becomes apparent how the many disciplines work in concert. The QA program will not presume that certain activities occur. It will require that documentations and controls be implemented and evaluated for effectiveness on a prescribed frequency basis. These evaluations describe deficiencies and corrective actions required.

In the formulation of the QA plan for Region V the mandates for carrying out QA are documented in the Quality Assurance Policy (Section 2). In this policy, management has designated responsibilities and the individual who bear those responsibilities. The organizational structure into which the QA management interacts is established by this policy (Appendix 2). Administratively, the QAO may be shown in a different relationship.

4.2.1 Assignment of Responsibilities

The Quality Assurance Office located in the Surveillance and Analysis Division has the responsibility of managing Region V's quality assurance program.

The Quality Assurance Office (QAO) establishes policies and guidelines for regional, state and local quality assurance programs, and conducts independent audits. Quality control, i.e., quality and documentation of data used by regional/state/local personnel, is the responsibility of the data generator. The mission of the QAO is to ensure through implementation of the quality assurance program so that the quality of data collected, reported or used by the Region is properly documented and that the data are sufficiently accurate and precise to meet Regional program needs. The Quality Assurance Office is responsible for developing and implementing procedures (programs) to insure the reliability of laboratory data supporting the air, pesticides, solid waste and toxic substances programs in the Air and Hazardous Materials Division, the public drinking water, ambient surface and ground water, and industrial and domestic wastewater programs in the Water Division; enforcement actions in the Enforcement Division; the International Joint Commission, the harbor dredging programs in the Great Lakes National Program Office, and all other programs generating environmental data for the Region.

QAO conducts annual on-site system evaluations. The evaluations are of the quality assurance and quality control programs of State laboratories and monitoring facilities that carry out testing under the Clean Air Act, Clean Water Act, Resource Conservation and Recovery Act, Safe Drinking Water Act and the Toxic Substances Control Act. In some instances local agencies are evaluated where state responsibility has been delegated. QAO identifies deficiencies, recommends corrective action and monitors effectiveness of action taken.

The QAO reviews state program plans for compliance with Agency requirements for quality assurance and analytical methodology used in laboratories and field operations. The QAO coordinates quality assurance programs with Agency regulations, program guidance and media strategy.

The on-going management of the laboratory certification program, pursuant to the Safe Drinking Water Act, is the responsibility of the QAO. This function also involves continued quality assurance activities for certified laboratories; an overview of state certification programs for certification of local laboratories and the performance of State certification officers.

The Quality Assurance Office manages an interlaboratory audit program which provides an extensive reference and quality control sample program for cooperating Federal, Canadian, State and local agencies, and private laboratories in Region V.

Approximately 123 laboratories participate in this program. Up to 316 different parameters are analyzed on a regular basis. The audits cover air, public water supply, ambient water (large lakes included), wastewater, dredging (sediments) and toxic pollutant laboratory analytical activity. These audits are extremely important for the determination of accuracy of laboratory performance. Results are evaluated and recommendations made for corrective actions for any deficiencies identified.

The management of the alternate test procedure program for compliance with the Safe Drinking Water Act, National Pollution Discharge Elimination System and other regulations, is the responsibility of the QAO. This function includes technical interepretation of the regulations relating to test procedures, coordination of applications, evaluation of applicants' technical data for equivalency and recommendations for approval or disapproval.

The QAO participates in quality assurance activities for the International Joint Commission Water Quality Board's monitoring activities on the Great Lakes. This function includes critical reviews of technical reports, maintenance of approved analytical methods and methods under consideration for approval, official interpretations of method equivalency for regulatory actions and defends regional data generated by approved procedures.

The QAO is responsible for providing, as requested, review and technical assistance concerning Agency analytical methodology and quality assurance requirements for State and local environmental agencies, NPDES dischargers, public water supplies, source emissions, etc. The QAO interprets National and Regional EPA policies in the areas of analytical methodology and quality assurance.

The QAO is responsible for the management of the quality assurance requirements for all Region V external projects involving collection and analytical measurements, which includes, but is not limited to, grants, contracts, cooperative agreements, and interagency agreements. The QAO's primary function is to insure that all analytical measurements conducted with Regional funding results in usable data of known quality that is acceptable for Region V's purposes. The air responsibility includes maintenance and primary calibration of field and laboratory equipment relative to air pollutants measurements, and step-by-step demonstrations of all facets of instrument maintenance, calibration and operation.

4.2.2. Flow of Information

The QAO is assigned activities under decision units which require evaluation of data producing systems. The S&A Director establishes priorities and delegates resources to the various tasks. These tasks are identified in the annual work plan. The QAO identifies goals to accomplish the objectives of the decision units per the specification from Headquarters program guidance from the Regional media programs, (for example, evaluation of QA programs for air monitoring in State Agencies). The QAO, through the S&A Director, establishes contact with State Agencies and arranges for information about the State's program and an on-site visit. Information obtained prior to an on-site visit is evaluated, the on-site evaluation is performed and an evaluation report is prepared. The evaluation is reported through the S&A Director to the State, to the Regional Air Program Office and the State Coordinators. Corrective action, deficiencies and recommendations are reported. States report corrective action taken or give reasons for not taking action to the QAO. Should the corrective action be of such a nature that an on-site visit is required to verify that the action was appropriate. A visit is requested through the S&A Director. The on-site visit is reported in a similar manner to the original evaluation report stating the findings and indicating satisfactory or non-satisfactory results and recommending future action as required.

When problems exist in which no correction is made and a dispute results, the findings with recommendations are reported to the Program Office and to the State Coordinators for resolution. If a State program is involved and the program office is unable to resolve the problem and an impasse is reached, the Regional Administrator makes a final determination of the unresolved issue based on recommendations from the QAO and program office. Basically two major types of reports are generated by the QAO. They are accuracy and performance audits and on-site system evaluation reports. The content of these reports are outlined in Section 8.7.2 and Subsection 8.7.2.1 of this document.

Based on the frequency identified in the QAO program plan (Section 12), the QAO will write interpretative reports to management. These reports will be made on a regular basis and will identify areas of work that could be improved and areas that are being performed properly or in an exceptional manner. These reports will be based on information obtained during on-site evaluations, from reviews of performance sample analyses and from evaluations of routine quality control audit data.

4.2.3 Identification of QA - Related Committees or Meetings

Quality assurance requirements/information are transmitted within Region V through meetings called by the QAO with affected Regional media personnel. These are on an as needed basis. Documentation is also provided by way of memoranda.

Quality assurance requirements/information are transmitted to State and local agency laboratory directors and quality assurance coordinators by written communcations from the Quality Quality Assurance Office on a as needed basis. QA information is also disseminated through the audit and on-site evaluation of Regional, State and local agency monitoring activities during the frequencies specified in the QAO's FY 80 program plan (Appendix 1).

The QAO will conduct two workshops in FY 80 for public water supply analysts. The workshops will be for standardizing metal analyses and for upgrading organic analyses for public water supply laboratories. The Central Regional Laboratory will provide technical support to the QAO in this endeavor by making their laboratory facilities available and having their personnel participate in the workshops.

The QAO has identified State and local air program agencies that are in need of technical assistance in the area of quality assurance and laboratory capability. Through a contract that EMSL-RTP has in place, the QAO will work with the contractor to upgrade those agencies that are in need of technical assistance. This activity will commence the 2nd quarter of FY 80.

A workshop for audits of air monitoring sites will be conducted by QAO and RTP at Region V in March 1980 for Region, State and local agency personnel engaged in auditing air monitoring sites.

The Quality Assurance Office participates in the Agency's regularly scheduled semi-annual QA coordinator's meeting where the Agency's QC concerns are addressed. The QAO participates in national short term QA tasks as requested by the National Program.

The Chief, QAO has been appointed to the Data Quality Work Group, Surveillance Sub-Committee, International Joint Commission, Water Quality Board. The Data Quality Work Group has the responsibility of assuring the quality of data from participating laboratories engaged in the Surveillance Sub-Committee's Great Lakes Surveillance Plan. All IJC QA activities are implemented through the Data Quality Work Group. The Work Group meets monthly.

4.2.4 Description of Needs

The following resources are required to accomplish the QA objectives and milestones identified in the interim QA program for Region V.

A. Staff - Twelve man years of effort is required to fully implement the Quality Assurance Program for Region V. Sixty-six (66) percent of staff is in place. Present staff consist of the Office Chief, 1 secretary, 2 professional chemists, 1 professional microbiologist, 1 professional physical scientist, 1 journeyman organic chemist and 1 journeyman electronics technician. Professional organic chemistry support is provided to the QAO on an as needed basis from the CRL (this support will continue for the "hands on" experience). The type of additional staff required (34%) is 1 professional organic chemist, 1 statistician (or chemist with a good statistical background) and 2 journeyman inorganic chemists.

B. Monetary - The QAO needs approximately $120,000 for contracts. These contracts are to be used to develop software EDP capability for measurement methods and statistical data evaluation for minimum turn around time. Data quality problems could be identified much faster and larger volumes of data can be evaluated.

C. Time - If all resources identified in Section 4.2.4 are granted the QAO, the program described in this document could be fully implemented in 90 days after receipt of resources.

D. Training Seminars - The QAO is providing workshops for standardizing metal analyses and upgrading organic analyses performance for Region V and State laboratory personnel during the second quarter of FY 80. A workshop for audits of air monitoring sites will also be provided Region V, State and local agency personnel. Travel funds will be required to get personnel to these workshops when their agency can not afford to send them. Approximately $2,000 is required for this travel.

5. PERSONNEL

Key personnel of the Quality Assurance Office must have sufficient administrative and technical stature to be considered a peer to the Managers of monitoring activities within the Region and to the Managers of Region V State and local laboratories. This staff must have a professional knowledge/training and understanding of chemical/microbiological principles, concepts, practices, established methodology and measurement (instruments) systems. The individual must have at least two years of bench experience in his/her speciality, particularly in an environmental laboratory. The individual must have experience in developing and implementing intralaboratory quality control programs. Regional QAO personnel must have knowledge of Federal laws, Agency regulations and guidelines pertaining to quality assurance and analytical procedures related to the Agency's regulatory monitoring programs. The individual must be experienced in meeting and dealing with Regional, State and local government officials and other Federal Agencies.

Analytical operations in the laboratory can be graded according to the degree of complexity. Some analyses require no sample treatment, and the measurement can be performed in minutes on a simple instrument. Other determinations require extensive sample preparation prior to complex instrumental examination. Consequently, work assignments in the laboratory should be clearly defined. Each analyst should be completely trained and should fully understand all the assignments of his job before being given new responsibilities. In this regard, all analysts, subprofessional or professional, should be thoroughly instructed in basic laboratory operations, according to the extent of professional maturity. Some of the basic operations that will be reviewed with laboratory personnel during the on-site evaluation follow.

a. SAMPLE LOGGING: Routine procedure for recording of samples entering the laboratory and assigning primary responsibility should be emphsized. The information that is required and the routing of the samples to the analyst is then established. The stability, preservation, and storage of samples prior to analyses are then discussed.

b. SAMPLE HANDLING: The analyst should understand thoroughly at which points in his procedures the sample is to be settled, agiated, pipetted, etc., before he removes it from the original container.

c. MEASURING: The analysts, especially new employees and subprofessionals, should be instructed in the use of volumetric glassware. The correct use of pipettes and graduates should be emphasized.

d. WEIGHING: Because alsmost every measuring operation in the analytical laboratory is ultimately related to a weighing operation, the proper use of the analytical balance should be strongly emphasized. Maintenance of the balance, including periodic standardization, should be repeatedly emphasized to all personnel.

e. GLASSWARE: All glassware should be washed and rinsed according to the requirements of the analysis to be performed. Not only must the personnel assigned to these tasks be instructed, but also all lab personnel should know the routine for washing and special requirements for particular uses of glassware. In addition, the precision tools of the the laboratory such as pipets, burets, graduates, and tubes should be inspected before use for cleanliness, broken delivery tips, and clarity of marking. Defective glassware should be discarded or segregated.

f. INSTRUMENTATION: Operation and maintenance of analytical instrumentation is of primary consideration in the production of valid data. All instruments must meet the requirements specified in Agency regulations, be properly calibrated, quality-control checks documented, and standard curves verified on a routine basis. References on instrumental quality control are presented in Section 8.4 and 8.5 of this document.

g. DATA HANDLING AND REPORTING: As with sample logging, the routine procedure for recording results of analyses and pertinent observations, including quality control checks, should be emphasized. Analytical data should be permanently recorded in meaningful, exact terms and reported in a form that permits future interpretation and unlimited use. Details are discussed in Section 9 of this document.

h. QUALITY CONTROL: The need to continuously assess precision and recovery values of methodology is a prime responsibility of the analyst. Self-evaluation through the analyses of QC samples, replicates and recovery of spikes from samples representative of the daily workload provides confidence and documentation of the quality of the reported data.

i. SAFETY: Laboratory safety should be discussed on a continuing basis with all employees, but it should be emphasized when an employee is assigned to perform new duties.

j. IMPROVEMENT: In summary, quality control begins with basic laboratory techniques. Individual operator error and laboratory error can be minimized if approved techniques are consistently practiced. To insure the continued use of good technique, laboratory supervisors should periodically review the basic techniques and point out areas of needed improvement with each analyst.

Continuing improvement of technical competence by all laboratory personnel is, of course, the final responsibility of the laboratory supervisor. In a well-organized laboratory, however, a big-brother attitude of higher ranking to lower grade personnel should be encouraged; each person should be eager to share experience, tricks of the trade, special skills, and special knowledge with subordinates. Obviously, efficiency and results will improve.

k. SKILLS: The cost of data production in the analytical laboratory is based largely upon two factors: the pay scale of the analyst, and the number of data units produced per unit of time. However, because of the large variety of factors involved, estimates of the number of measurements that can be made per unit of time are difficult. If the analyst is pushed to produce data at a rate beyond his capabilities, unreliable results may be produced. On the other hand, the analyst should be under some compulsion to produce a minimum number of measurements per unit of time, lest the cost of data production become prohibitive. In table 5-1, estimates are given for the number of determinations that an analyst should be expected to perform on a routine basis. The degree of skill required for reliable performance is also indicated.

The time limits presented in the table are based on use of approved methodology. A tacit assumption has been made that multiple analytical units are available for measurements requiring special equipment, as for cyanides, phenols, ammonia, nitrogen, and COD. For some of the simple instrumental or simple volumetric measurements, it is assumed that other operations such as filtration, dilution, or duplicate readings are required; in such cases the number of measurements performed per day may appear to be fewer than one would normally anticipate.

6. FACILITIES, EQUIPMENT, AND SERVICES

The QA program makes Facilities, Equipment and Services a major component of the program. The recognition is made that no data can be collected without the appropriate equipment that is functional. To assure the operation of that equipment all facilities, equipment and services must work as a composite in a smooth orderly manner. The items that are necessary are:

A. Laboratory facilties, building, utilities, equipment and maintenance.

B. Field facilities, housing for equipment, transportation requirements, utilities, supplies, communications and maintenance.

C. Analtytical equipment, required methods, operation and calibration manuals, maintenance, parts and supplies.

D. Procurement procedures, that require purchase of the required equipment with warrantees, demonstrated satisfactory performance prior to payment, service arrangements, availability of spare parts, evaluation of equipment from information of prior users, costs evaluation and comparisons against competitive equipment.

TABLE 5-1
SKILL-TIME RATING OF STANDARD ANALYTICAL OPERATIONS

Measurement	Skill Required (Rating No.)[1]	Number Per Day
Simple Instrumental:		
pH	1	100-125
Conductivity	1	100-125
Turbidity	1	75-100
Color	1	60-75
Dissolved Oxygen (Probe)	1,2	100-125
Fluoride (Probe)	1,2	100-125
Simple Volumetric:		
Alkalinity (Potentiometric)	1	50-75
Acidity (Potentiometric)	1	50-75
Chloride	1	100-125
Hardness	1	100-125
Dissolved Oxygen (Winkler)	1,2	75-100
Simple Gravimetric:		
Solids, Suspended	1,2	20-25
Solids, Dissolved	1,2	20-25
Solids, Total	1,2	25-30
Solids, Volatile	1,2	25-30
Simple Colorimetric:		
Nitrate N (Manual)	2	75-100
Nitrate N (Manual)	2	40-50
Sulfate (Turbidimetric)	2	70-80
Silica	2	70-80
Arsenic	2,3	20-30
Complex, Volumetric, or Colorimetric:		
BOD	2,3	[2]15-20
COD	2,3	25-30
TKN	2,3	25-30
Ammonia	2,3	25-30
Phosphorus, Total	2,3	50-60
Phenol (Distillation Included)	2,3	20-30
Oil and Grease	2,3	15-20
Fluoride (Distillation Included)	2,3	25-30
Cyanide	2,3	8-10
Special Instrumental:		
TOC	2,3	75-100
Metals (by AA), No Preliminary Treatment	2,3	150
Metals (by AA), With Preliminary Treatment	2,3	60-80
Organics (by GC), Pesticides, Without Cleanup	3,4	3-5
Organics (by GC), Pesticides, With Cleanup	3,4	2-4

[1] Skill-required rating numbers are defined as follows:

1 - aide who is a semiskilled subprofessional with minimum background or training, comparable to GS-3 through GS-5.

2 - aide with special training or professional with minimum training with background in general laboratory techniques and some knowledge of chemistry, comparable to GS-5 through GS-7.

3 - experienced analyst capable of following complex procedures with good background in analytical techniques, professional, comparable to GS-9 through GS-12.

4 - experienced analyst specialized in highly complex procedures, professional, comparable to GS-11 through GS-13.

[2] Rate depends on type of samples.

E. Preventive Maintenance policies and procedures.

F. Service and Repair procedures.

G. Audit and Evaluation requirements.

H. Safety.
A protocol for procurement testing is described in Appendix 4 which establish guidelines for equipment that are based on EPA guidelines, good laboratory practices and pertinent information from industries and governmental agencies where similar concerns are part of the art of good management and quality assurance. As with other operations, the effectiveness of facilities are determined by independent evaluations.

7. REVIEW OF PROGRAM PLANS, PROJECT PLANS, OR STUDY PLANS

As a statement of policy, the QA program requires a review of all program project and study plans for Region V, including the S&A Division study plans. It is essential that these plans are evaluated from the beginning so that the appropriate measurement method is selected that will produce the data the user needs. Many, if not all, projects require data that lead to decisions that have an economic impact as well as technologic impact. The prevention of loss in monies, resources and time weighs heavily upon the plans that lead to program or project development. If those plans incorporate unapproved, and inappropriate methodology which in turn produce data that are not pertinent to the program or project or do not have acceptable precision, accuracy, representiveness or completeness, then the efforts are lost, lead to wrong decisions, or cause equivocation.

Since the review of plans has not been customary in the past, it will be necessary to develop programs that accomplish this preliminary review process. The various divisions and program units and QA must work together to initiate this process as a Standard Operating Procedure. The details that need to be accomplished are:

A. Directives to Program, Project or study offices requiring QA review of scope and plans at the earliest data of the planning process.

B. Request of QA review requirements must be in writing. This request should give some estimate of the magnitude of study.

C. QAO, immediately upon initiation of the review process, would:
   1. determine expertise required
   2. technology

D. Having established plans for review, the QA review would proceed and results would be reported. The evaluation would declare feasibility with respect to provisions that assure the appropriate methodology, precision, accuracy, representativeness and completeness. Should factors be inappropriate or deficient, corrective measures would be stated to the project officer.

The Agency's protocol for evaluation of QA plans in extramural projects and contracts will be used as soon as the document is available.

QAO will investigate the needs for developing guidelines which would be used to evaluate statistical, modeling, and other aspects of environmental studies. The location of expertise and at times technology for unusual projects will require national concern. Thus these needs will be formulated as they become appropriate.

A review of programs, projects or study plans would determine what QA plans are to be incorporated in those plans. The various activities and items that must be identified are:

o Staffing (personnel in numbers, qualifiication and training).

o Methods (EPA approved methods must be used where required). Procedures must be documented and made available for review prior to use.

o Quality control measures must be described in detail. This would explain the frequency of duplicate, spike or performance samples. Control measures used in sample collection, with frequency of duplicate sampling prescribed. Audits by interlaboratory, peer group, systems audits and performance audit must be described as to frequency and source of audit. Control limits must be determined and the required measures that must be taken when out of control limits have been exceeded should be described.

o Sample collection and preservation should be described in detail. The calibrations of analytic methods and equipment must be according to the requirements of the approved methods. Standards used for calibrations must be of the highest purity and referenced to NBS standards whenever possible. Calibration procedures and tracability must be documented.

The QA plans must appear early in the planning process because without these QAO will not have suitable information to move forward in the evaluation process.

Suitable procedural information for developing QA plans based on the items discussed above are available in the references cited in Section 14 of this document.

8. DATA COLLECTION

Data quality changes occuring during data collection can come from six major activities: a) formulating sound objectives for the sampling program, b) collecting representative samples, c) maintaining sample integrity through proper sample handling and preservation, d) adhering to appropriate sample identification and, where needed, chain of custody procedures, e) practicing quality assurance procedures in the sample transportation, storage, and preparation processes, and f) using proper analytical techniques complete with appropriate quality control activities to generate the actual data.

8.1 Sampling Plan

The objectives of the sampling program affect all the other aspects of the sampling program. Sampling program objectives are determined by the following activities: (a) planning (areawide or basin), (b) permits, (c) compliance, (d) enforcement, (e) design, (f) process control, and (g) research and development. The types of sampling programs to be employed, depending on suitability to program objectives, include reconnaissance surveys, point-source characterization, intensive surveys; fixed-station-network monitoring, ground-water monitoring, ambient air monitoring and stationary source emission monitoring, and special surveys involving chemical, biological, microbiological, and radiological monitoring.

Factors that must be considered in meeting the objectives of the sampling program are the extent of the manpower resources, the complexity of the parameters of interest, the duration of the survey, the number of samples, the frequency of sampling, the type of samples (grab or composite), and the method of sample collection (manual or automatic).

The media activity will identify the need for a sampling activity in Region V. A person with lead responsibility in the media activity is also identified to coordinate the project for the media activity.

The identified need is transmitted to the S&A Division. The Technical Support Branch coordinates the formulation of objectives and goals for the sampling activity with the Central Regional Laboratory and the appropriate District Office. Once goals have been formulated to accomplish objectives (including the six major activities listed under 8 above), the proposal is then reviewed by the QAO to insure that all quality assurance requirements for producing valid data have been included. If any QA changes are needed, the QAO will specify the changes needed. Once the QA changes are made (if need be), the QAO will concur. The S&A Division Director will transmit the study proposal to the Director of the requesting media program for review and see if the defined objectives and goals meets the program needs. If not, revision will be made (NOTE - QA is not to be compromised). Once the Director of the media program concurs in the proposal, the appropriate S&A Division Office/Branch or other Divisions will initiate the proposal.

8.2 Sampling Methodology

The objective of sampling is to obtain a representative portion of the total environment under investigation. The sampling plan shall contain, as a minimum, the following factors for concurrence by the QAO (Item 8.1 above) in formulation of the sampling plan.

A. Water and Wastewater

o Site Selection

The location of the sampling site is critical in obtaining representative data. Preferably, water sampling sites for point sources of pollution from municipal and industrial effluents are located at points of highly turbulent flow to insure good mixing; however, inaccessibility, lack of site security, or power unavailability may preclude use of the best sites, but these impediments should not be used as reasons for collecting samples at unacceptable locations. Loctions of sampling sites for streams, lakes, impoundments, estuaries, and coastal areas vary, but in general occur in the following bodies: (a) in water bodies for sensitive uses (swimming and drinking water supply), (b) in major impoundments or reservoirs near the mouths of major tributaries and in the rivers entering and leaving the impoundments, (c) in water bodies polluted by man's activities, (d) in rivers upstream and downstream from tributaries, and (e) where hydrological conditions change significantly.

o Sample Type

The basic types of water and wastewater methods are grab sampling and composite sampling. Composite sampling may be conducted manually or automatically. The six methods for forming composite samples, all of which depend on either a continuous or periodic sampling mode, are the following: (a) constant sample pumping rates, (b) sample pumping rates proportional to stream flow rates, (c) constant sample volumes and constant time intervals between samples, (d) constant sample volumes and time intervals between samples proportional to stream flow rates, (e) constant time intervals between samples and sample volumes proportional to total stream flow volumes since last sample, and (f) constant time intervals between samples, and sample volumes proportional to total stream flow rates at time of sampling. The choice of using the grab sampling method or one of the six compositing sampling methods is determined by program objectives and the parameters to be sampled.

o Use of Automatic Samplers

The use of automatic samplers eliminates errors caused by the human element in manual sampling, reduces personnel cost, provides more frequent sampling than practical for manual sampling, and eliminates the performance of routine takes by personnel. Criteria for brand selection of automatic samplers include evaluations of the intake device, intake pumping rates, sample transport lines, sample gathering systems (including pumps and scoops), power supplies and power controls, sample storage systems, and additional desirable features to fit particular sampling conditions. There are many commerically available automatic samplers; however, because no single automatic sampler is ideally suited for all situations, the user should carefully select the automatic sampler most suited for the particular water or wastewater to be characterized. Precautions must be taken in regard to using certain types of samples in potentially explosive atmospheres.

o Flow Measurements

An essential part of any water or wastewater sampling survey as well as a necessary requirement of the National Pollution Discharge Elimination System (NPDES) permit program is accurate flow measurements. Flow measurement data may be instantaneous or continuous.

For continuous measurements, a typical system consists of primary devices such as weirs and flumes and secondary devises such as flow sensors, transmitting equipment, recorders, and totalizers. The improper installation or design of a primary device or malfunction of any part of a secondary device results in erroneous flow data. The accuracy of flow measurement data also varies widely, depending principally on the accuracy of the primary device and the particular flow measurement method used. In any case, measurements should be within ±10 percent of the true values.

As part of a monitoring activities' QA program, a written step-by-step procedure for the use of each type of flow equipment employed by the monitoring activity shall be available. The write-up is to include the protocol for installation of the measuring device (if appropriate), maintenance and verifiction of calibration of the measuring device in the field. Documentation must also be maintained. All mechanical and electronic type current meters' calibration are to be traceable through an unbroken chain (supported by documentation to some untimate or national reference standard (i.e., NBS or NOAA).

o Statistical Approach to Sampling

Four factors must be established for every sampling program: (a) number of samples, (b) frequency of sampling, (c) parameters to be measured, and (d) sampling locations. These factors are usually determined in varying degrees by details of the pertinent discharge permits or are more arbitrarily set by the program resource limitations. Nevertheless, the nature of the statistical methods selected and scientific judgment should be used to establish the best procedures.

- Special Sampling Procedures

  Special sampling procedures should be employed for hazardous wastes, toxics, municipal, industrial, and agricultural waters, and surface waters as well as bottom sediments and sludges, and for biological, microbiological, and radiological studies.

B. Air

- Sampling Site Selection Considerations

The need for an air quality monitoring program usually is related to one or more of the following objectives:

1. To judge compliance with and/or progress made toward meeting ambient air quality standards.

2. To activate emergency control procedures that prevent or alleviate air pollution episodes.

3. To observe pollution trends throughout a region, including nonurban areas.

4. To provide a data base for research evaluation of effects; urban, land use, and transportation planning; development and evaluation of abatement strategies; and development and validation of diffusion models.

Sampling site and equipment requirements are generally divided into three categories, consistent with desired averaging times:

1. Continuous--Pollutant concentrations determined with automated methods and recorded or displayed continuously.

2. Intermittent--Pollutant concentrations determined with manual or automated methods from integrated hourly or daily samples on a fixed schedule.

3. Static--Pollutant estimates or effects determined from longer-term (weekly or monthly) exposure of qualitative measurement devices or materials.

Air quality monitoring sites that employ automatic equipment to continually sample and analyze pollutant levels may be classified as primary. Primary monitoring stations are generally located in areas where pollutant concentrations are expected to be among the highest and in the areas of highest population density and, as such, are often employed in health effects research networks. In addition, these stations are designed as a part of the air pollution episode warning system.

o Network Design Considerations

In designing an air quality monitoring activity, the following four criteria for locating sites should be considered, either singly or in combination, depending upon the objective of sampling:

1. Orient monitoring sites to measure the impacts of known pollutant emission categories on air quality.
2. Orient monitoring sites relative to population density to measure receptor-dose levels, both short and long-term.
3. Orient monitoring sites to measure the impacts of known pollutant emission sources (area and point) on air quality.
4. Orient monitoring sites to obtain measurements representative of areawide air quality.

In order to select locations according to these criteria, it is necessary to have detailed information of the location of sources of emission, the geographical variability of ambient pollutant concentrations, meteorological conditions, and population density.

o Representative Sampling

Assuring the collection of a representative air quality sample depends on the following factors:

1. Locating the sampling site and determining network size consistent with monitoring objectives.
2. Restraints on the sampling site imposed by meteorology.
3. Restraints on the sampling site imposed by local topography, emission sources, and physical constraints.
4. Sampling schedules consistent with monitoring objectives.

o Locate Sampling Site and Determine Network Size

Consistent with monitoring objectives previously noted, networks are designed to meet at least one of four major objectives. The following tabulation presents examples of currently implemented networks applicable to each of these "objectives" categories:

Objective	Network	Comment
Compliance monitoring	SIP (State Implementation Plan)	To demonstrate attainment or maintenance of Air Quality Standards
Emergency episode monitoring	SIP/local agency emergency control program	To activate immediate, short-term, emission controls for prevention of episodes
Trend monitoring	NASN (National Air Sampling Networks)	To fulfull mandate of Federal legislation
Research monitoring	CHAMP (Community Health Air Monitoring Program)	To determine long-term pollutant trend in selected areas with respect to health effects

o Compliance Monitoring

The information required for selecting sampler location is essentially the same as that for determining the number of samplers, i.e., isopleth maps, population density maps, and source locations. Following are suggested guidelines:

1. The priority area is the zone of highest pollutant concentration within the region. One or more stations are to be located in this area.
2. Close attention should be given to densely populated areas within the region, especially when they are in the vicinity of heavy pollution.

3. For assessing the quality of air entering the region, stations must also be situated on the periphery of the region. Meteorological fctors such as frequencies of wind direction are of primary importance in locating these stations.

4. For determining the effects of future development on the environment, sampling should be undertaken in areas of projected growth.

5. A major objective of surveillance is evaluation of progress made in attaining the desired air quality. For this purpose, sampling stations should be strategically situated to facilitate evaluation of the implemented control tactics.

6. Some information of air quality should be available to represent all portions of the regions.

Some stations will be capable of fulfilling more than one of the functions indicated; e.g., a station located in a densely populated area can indicate population exposures and also document the changes in pollutant concentrations resulting from control strategies employed in the area.

o Emergency Episode Monitoring

For episode avoidance purposes, data are needed quickly--in no less than a few hours after the sensor is contacted by the pollutant. While it is possible to obtain data rapidly by on-site manual data reduction and telephone reporting, there is a trend toward automated monitoring networks. Obviously, the severity of the problem, size of the receptor area, and availability of resources influence both the scope and sophistication of the system.

It is necessary to utilize continuous air samplers because an episode lasts only a few days and the control actions taken must be based on "real-time" measurements correlated with the decision criteria. Based on alert criteria now in use, 1-hour averaging times are adequate for surveillance of episode conditions. Shorter averaging times provide information on data collecting excursions but increase the need for automation because of the bulk of the data obtained. Averaging times longer than six hours are not desirable because of the delay in response this imposes.

Collection and analysis must be accomplished rapidly if the data are to be useful immediately. There is no time to check out the methods, run blanks, calibrate, etc., after the onset of episode conditions. In order for the instrument to be maintained in peak operating condition, personnel must be stationed at the sites during the episode or automated equipment must be operated that can provide automatic data transmission to a central location.

Episode conditions threaten human welfare, and monitoring sites should be located in areas where this welfare is most threatened:

1. In densely populated areas.

2. Near large stationary sources of pollutants.

3. Near hospitals.

4. Near high-density traffic interchanges.

5. In homes for the aged.

A network of sites is useful in determining the range of pollutant concentrations within an area. Although the most desirable monitoring sites are not necessarily the most convenient, consideration should be given, for reasons of access, security, and existing communications, to the use of public building: schools, firehouses, police stations, hospitals, and water or sewage plants.

o Trend Monitoring

As typified by the National Air Surveillance Network (NASN), trend monitoring is characterized by locating a minimal number of monitoring sites across as large an area as possible. The program objective is to determine, in a broad sense, the extent and nature of air pollution as well as determine the variation in the measured levels of atmospheric contaminants in respect to geographic, socioeconomic, climatologic and other factors. The data acquired are useful in planning epidemiological investigations and also provide the background against which more intensive community and state-wide studies of air pollution can be conducted.

Urban sampling stations are usually located in the most densely populated areas of a region. In most regions there are several urban sites.

Nonurban station loctions include various topographical categories such as farmland, desert, forest, mountain, and coastal. The nonurban stations are not specifically selected to be "clean air" control sites for urban areas, but they do provide for a relative comparison between some urban and nearby nonurban areas.

In interpreting trend data one must consider the limitations imposed by the network design. Even though precautions are taken to ensure that each sampling site is as representative as possible of the designated area, it is impossible to be totally certain that the measurements obtained at a specfic site are not sometimes unduly influenced by local factors. Such factors might include topography, structures, and sources of pollution in the immediate vicinity of the site, and other variables, the effect of which cannot always be accurately anticipated but which should be considered in network design. It must be kept in mind that when comparisons are made among pollution levels for various areas, they are valid only insofar as the sites are comparable.

o Research Monitoring

An example of a research-oriented air quality monitoring effort is the EPA's Community Health Air Monitoring Program (CHAMP), which is providing data to develop criteria for both short- and long-term air quality standards. Air monitoring networks related to health effects are composed of integrating samplers for determining pollutant concentrations for 24 hours, or longer for developing long-term ($\geq$ 24 hours) ambient air quality standards. These studies require that monitoring points be located so that the resulting data represent the population group under study. The monitoring stations are therefore established in the center of small, well-defined residential areas within a community. Data correlations are made between observed health effects and observed air quality exposure.

Requirements for aerometric monitoring in support of health studies are:

1. Station must be located in or near the population under study.

2. Pollutant sampling averaging times must be sufficiently short to allow for use in acute health effects studies that form the scientific basis for short-term standards.

3. Sampling frequency should be sufficient to characterize air quality as a function of time, usually daily.

4. System should be flexible and responsive to emergency conditions with data available on short notice.

o Meterological Factors that Affect Representative Sample Collection

Meteorology must be considered in determining not only the geographical location of a monitoring site, but also such factors as height, direction and extension of sampling probes. Meteorological parameters having the greatest influence on dispersion of pollutants are the direction, speed, and variation of wind.

Wind direction provides an indication of the general movement of pollutants in the atmosphere. Review of available data can indicate mean wind direction in the vicinity of the major sources of emissions.

The effects of wind speed are two-fold. First, wind speed determines the travel time from source to receptor. Second, wind speed affects dilution in the downwind direction, i.e., concentration of air pollutants is inversely proportional to wind speed.

o Topographical Features that Affect Representative Sample Collection

The transport and diffusion of air pollutants is complicated by topographic features. Minor topographic features may exert small influence; major features, such as deep river valleys or mountain ranges, may affect large areas. Before final site selection, topography of the area should be reviewed to ensure that the purpose of monitoring at that site will not be adversely affected.

Final placement of the monitor at a selected monitoring site depends on physical obstructions and activities in the immediate area, accessibility, availability of utilities and other support facilities, correlation with the defined purpose of the specific monitor, and monitor design. Because obstructions such as trees and fences can significantly alter air flow, monitors should be removed from such obstructions. It is important that air flow around the monitor should be representative of the general air flow in the area to prevent sampling bias.

Network designers are to avoid sampling locations that are unduly influenced by down-wash or by ground dust, such as a rooftop air inlet near a stack or a ground-level inlet near an unpaved road. In the latter case, either elevate the sampler intake above the level of maximum ground turbulence effect or simply place it reasonably far from the source of ground dust.

o Sampling Schedules Consistent with Monitoring Objectives

Current Federal regulations specify the frequency of sampling for criteria pollutants to meet minimum SIP surveillance requirements. Continuous sampling is specified except for 24-hour measurements of total suspended particulate matter and 24-hour integrated values for SO_2 and NO_2. The high-volume and gas impinger measurements are required at least once every six days, equivalent to about 61 random samples per year. Twenty-four-hour samples should be taken from midnight (local standard time) to midnight and thus represent calendar days to permit direct utilization of the sampling data with standard daily meteorological summaries.

o Sample Preservation and Holding Times

During and after collection, if immediate analysis is not possible, the sample must be preserved to maintain its integrity. Proper handling of the samples helps insure valid data; consideration must also be given to care of the field container material and cap material, cleaning, structure of containers, container preparation for determination of specific parameters, container identification, and volumes of samples.

Sample collection containers, preservatives and holding times for samples collected in the 106, 208, 404(b), 1412 and the Great Lakes National Monitoring Programs shall be those specified in Appendix 5.

Sample collection, containers and preservation of industrial effluents for priority pollutants protocol shall be those specified in Appendix 6.

Sample collection and preservation protocol for hazardous waste samples shall be those specified in Appendix 7.

Sample collection and preservation protocol for ambient air samples shall be those specified in Appendix 8.

Sample collection, preservation and holding time protocol for the 1412 monitoring (public water supply) program shall be those specified in Appendix 14.

8.3 Analytical Methodology

The analytical laboratory provides qualitative and quantitative date for use in decision making. To be valuable, the data must accurately describe the characteristics and concentrations of constituents in the samples submitted to the laboratory. In many cases, because they lead to faulty interpretation, approximate or incorrect results are worse than no results at all.

Many analytical methods for environmental pollutants have been in use for many years and are used in most environmental laboratories. Widespread use of an analytical method in environmental testing usually indicates that the method is reliable, and therefore tends to support the validity of the reported test results. Conversely, the use of little-known analytical techniques forces the data user to rely on the judgment of the laboratory analyst, who must then defend his choice of analytical technique as well as his conclusions.

Uniformity of methodology within a single laboratory as well as among a group of cooperating laboratories is required to remove methodology as a variable when there are many data users. Uniformity of methodology is particularly important when several laboratories provide data to a common data bank (such as STORET) or cooperate in joint field surveys. A lack of uniformity of methodology may raise doubts as to the validity of the reported results. If the same constituents are measured by different analytical procedures within a single laboratory, or by a different procedure in different laboratories, it may be asked which procedure is superior, why the superior method is not used throughout, and what effects the various methods and procedures have on the data values and their interpretations.

Physical and chemical measurement methods used in environmental laboratories should be selected by the following criteria:

a. The selected methods should measure desired constituents or environmental samples in the presence of normal interferences with sufficient precision and accuracy to meet the environmental data needs.

b. The selected procedures should use equipment and skills ordinarily available in the average environmental laboratory.

c. The selected methods should be sufficiently tested to have established their validity.

d. The selected methods should be sufficiently rapid to permit repetitive routine use in the examination of large numbers of water samples.

The restriction to the use of EPA methods in all laboratories providing data to EPA permits the combination of data from different EPA programs and supports the validity of decisions made by EPA.

The QAC requires that the methodology be carefully documented. In some reports it is stated that a standard method from an authoritative reference was used throughout an investigation, when close examination has indicated, however, that this was not strictly true. Standard methods may be modified or entirely replaced because of recent advances in the state of the art or personel preferences of the laboratory staff. Documentation of measurement procedures used in arriving at laboratory data should be clear, honest, and adequately referenced; and the procedures should be applied exactly as documented.

Reviewers can apply the associated precision and accuracy of each specific method when interpreting the laboratory results. If the accuracy and precision of the analytical methodology are unknown or uncertain, the data user may have to establish the reliability of the result he or she is interpreting before proceeding with the interpretation.

As part of any monitoring program's quality control program, the analytical methodology must be included for review and approval by the Quality Assurance Office. The format and minimum requirements for method documentation are listed below:

1. Parameter that the method measures.

2. Principle - A brief description of the method.

3. Optimum Concentration Range - The analytical range from the lowest concentration to the highest concentration in which a substance is measured. The sample may be concentrated or diluted so that the substance can be detected within this range.

4. Sensitivity - The slope of a curve of concentration versus instrument response (such as absorbance).

5. Detection Limit - The lowest quantity which may be be distinguished from zero with an acceptable degree of confidence.

6. Reference - The source of the analytical method. In addition all variances of the original procedure are documented here.

7. Matrix - The general composition of the sample that the method is capable of handling, e.g., water (potable, ambient, wastewater), solids (leachates, sediments, sludges), air (filter particulates, bubbler solutions, casette trap). Fluids (solvents, hydrocarbons, oils).

8. Analysis Procedure
    a. Description - The analytical procedure is described for normal conditions. Sample pretreatment (if required) and preparation protocols are also described here. The language used to describe the method is to be detailed enough (cookbook fashion) so that a technician with experience in the respective type of analysis would clearly understand every step of the procedure. Analytical techniques that employ a great deal of instrumentation such as atomic absorption and automated analysers are briefly described since instrument manuals are available which detail the use of the instrument. However, auto analyzer manifolds are to be depicted.

b. Instrument parameters - A description of the instrument and all the instrument settings that are necessary to setup the instrument for normal conditions.

c. Routine performance tests - A test of the instrument performance which is separate from a calibration procedure, and is a gross indication of the instrument's response. This test is performed and documented each time a batch of samples is processed or else on a daily basis. The frequency chosen for instrument response check is dependent on the analysts' confidence of instrument stability.

d. Calibration standards - The calibration standards are described in terms of the range of concentrations used in the normal procedure and in terms of composition (preparation of standard solutions) employed for various matricies.

e. In-house quality control standards - There are standards which are different from calibration standards. Quality Control standards are meant to be a control procedure by which to judge whether the procedure is in-control or out-of-control after the various instrument checks have been satisfied. Wherever used, at least one quality control standard is determined with each batch of samples. The information is then documented.

   1. one wheel of samples - for auto analyzer techniques.

   2. a number of samples that is determined continuously without an interuption such as a coffee or lunch break or a change of instrument settings - for atomic absorption techniques, manual techniques, and gas chromatographic techniques.

f. Data calculations - Describe the computations and manipulations that must be used to convert raw data to a final analytical results.

g. Instrument log book - An indication of where the instrument log book is located. The instrument book is to contain, as a minimum, the following sections:

1. Name, Model Number, Serial Number
2. Does the identification verify that the instrument is EPA approved, if required
3. Instrument history
4. Service record
5. Routine performance test - This section includes a space for the date, initials of analyst, comments, and other instruments parameters if applicable.

9. Interferences - When interferences are suspected or indicated by other tests, the specific procedures for dealing with these interferences are described here.

10. Precision and Accuracy
The statistical precision and accuracy results for the parameter generated by the laboratory are to be documented.

11. Quality Control
    a. Internal Quality Control - In-House Quality Control Standards, in addition to being controls, are to be used as a measure of precision under ideal conditions. Frequency of use is to be specified. Reagent Blanks are to be determined to collectively check for possible contamination from the sample container, preservative, glassware, and laboratory reagents. Frequency of use is to be specified. The use of replicate analysis of real samples to measure precision is viewed as a product of the laboratories. This information is meant for use in interpreting analytical results and is of some use to the laboratory for evaluating the reported detection limits and detecting possible interferences that might not be documented in the original method. The use of replicates is dependent on the parameter (the number of samples with positive values) and the analytical method. Frequency of use is to be specified. The use of real sample spikes (positive

or negative) is also dependent on the convenience with which they fit into the analytical procedure. Spike are useful for evaluating recovery in addition to precision. Frequency of use is to be described.

b. External Quality Control - Participation in various comparative analytical programs and frequencies outside of the laboratories are cited here.

8.3.1 Maintenance of Up-To-Date File of Measurement Methods
The Central Regional Laboratory (CRL) currently uses over 200 approved reference methods to analyze over 500 different environmental pollutants. It is known from recent on-site inspections that the nine principal State laboratories in the Region use many analytical methods not used by the CRL and that they have made "minor" variations in methods in common use by EPA. The variety of new methods in use by the other Federal and local laboratories is not yet known, but it is expected that the total number of agency approved laboratory methods the QAO will be evaluating will number well over 1,000.

In addition to the laboratory methods, the QAO must monitor the performance of sampling procedures used by the monitoring programs conducted by Region V and a wide variety of field measurements. These include measurement of the common water parameters such as temperature, flow, dissolved oxygen, pH, etc., as well as the measurement of air pollutants using both continuous monitors and grab sampling techniques.

Each of the above methods must be technically evaluated and a decision made for each method as to whether or not the method is legally approvable for use in one or more of the many programs administered by EPA. For example, is the inductively coupled argon plasma procedure used by the CRL to analyze for metals significantly different from the approved atomic absorption procedure to prohibit it use in the analyses of public drinking water samples. A conservation QAO opinion would answer yes and therefore require the CRL to either use the manual method or obtain an alternate test procedure approval pursuant to the Safe Drinking Water Act. Either action would require at least 0.5 man years of effort which clearly identifies the importance of making the correct decision for each method-program combination. It should be again emphasized that the QAO program requires that each approved measurement method contain a complete description of all quality control audit procedures and the frequency and control limits to be used to insure reliability of reported data. Therefore, when a method is approved, the quality control program associated with that method will also be approved.

In summary, this function is not a "bookkeeping" job and is probably the most difficult work performed by the QAO. It requires in-depth technical and program skills as well as a great deal of organizational ability and diplomacy to negotiate satisfactory resolutions to the many problems currently facing the QAO in this area. QAO's initial approach toward completing this task is described below.

1. Program Guidelines and Implementation Plans
    a. The Reference Methods described in the various EPA regulations will serve as the basis for all method evaluations. Results obtained using the reference methods will be taken as the officially correct results even though it is known the result may not always accurately measure the contaminant concentration of interest.

       EPA offical analytical methodology for water quality measurements are given in Appendix. 9. Radiation methods are shown in Apendix 10. Ambient air measurement methods are shown in Appendix 11. Source air measurements analytical methodology are shown in Appendix 12. Public water supply measurements analytical methodology are shown in Appendix 13. Recommended analytical methodology for priority pollutants is referenced in Appendix 6. Sample preparation and analysis for hazardous waste are those specified in Appendix 7.

       Analytical measurements for ecological evaluations of proposed discharge of dredged or fill material into navigable waters are listed in Miscellaneous Paper D-76-17, titled Interim Guidance for Implementation of Section 404(b)(1) of Public Law 92-500 (FWPCA Ammendments of 1972), compiled by the U.S. Corps of Engineers.

    b. A unique number will be given to each method as it is approved. This will permit the QAO to more easily use computers to quickly retrieve information related to the method. The unique number will contain the following intelligence.

       o Laboratory using method

       o Sample type (air, water, sediment, biological)

- Parameter class (biological, organic, inorganic)
- Parameter (zinc, aldrin, TSP)
- Reference or alternate method
- EPA programs method may be used to support

c. Software programs will be written which will associate other information with each approved method using the method number as a cross index. Some of the related information will be as follows:

- QAO file folder where the "official" method description is maintained.
- A list of literature references supporting the method.
- The STORET, SARAD, etc., numbers related to the method.
- The appropriate reference method if the method is an alternate test procedure.
- All quality control audit data.
- A comment space for user remarks pertaining to method performance.

d. A cross-indexing system will be established in which one can obtain a list of approved laboratory-method combinations for each EPA program and a list of programs for which each laboratory is approved to use each of its described analytical methods.

e. All approved methods will be reviewed at least once each year (January - March) to insure that they are properly classified relative to any regulatory or technical changes thay may have occurred during the year.

f. A numerical description of the performance of each method will be obtained from the group evaluating the interlaboratory quality control audit data. These numbers will be evaluated, interpreted and a description will be prepared explaining the performance of the method for non-technical personnel. It will provide references to related methods.

g. The out-puts will be:

- An up-to-date list of all approved and some unapproved (those being processed, but not formally approved) methods for making any measurement. Each of these methods will have a list of programs which may be used to support the reference method, the proper number to use for storage of results (STORET, SARAD), the units of measure, the method performance data (detection limit, working concentration range, precision and accuracy) and laboratories approved to use the method.
- A copy of any approved method(s) and any approved quality control program(s).
- An evaluation of methods which were not approved for use by the QAO in the proposed program with justification for non-approval.

h. Relationship to other QAO functions
These official measurement methods will form the foundation of the QAO program for monitoring data reliability. They will be the "contract agreement" between the QAO and all media offices and will provide the written communication link for use in legal and technical challenges. They will provide a management structure for evaluating and documenting differences in method and laboratory performances resulting from "minor" changes in analytical methods and laboratory operating procedures.

8.3.2. Alternate Test Procedure Program
The Code of Federal Regulations (40 CFR 136, 40 CFR 35, 40 CFR 141, etc.), specifies that specific analytical methods be used to monitor compliance with several regulations administered by EPA. In each instance, the regulations provide a mechanism by which an alternate analytical procedure can be used in place of the specified reference procedure if it is first documented that the proposed alternate procedure is equivalent or better than the reference method. Unfortunately,

the different EPA programs specify different analytical mthods to be used to analyze for a given contaminant and different mechanisms for obtaining approval to use an alternate test procedure. This requires EPA to maintain records for all programs (NPDES, SDWA, etc.)-method (Flame AA, ICAP, Flameless AA, etc.)-laboratory combinations to insure that reported data can be used for regulatory purposes.

For Region V, the QAO is responsible for processing all alternate test procedure applications in the water program areas. Department E, EMSL-RTP, Research Triangle Park, North Carolina, has sole national responsibility for implementing designated reference and equivalent methodologies for the air programs as specified by 40 CFR 53.1. The Region V alternate test procedure protocols are described below by program.

8.3.2.1 Elements of an Application for a National Pollutant Discharge Elimination System (NPDES) or Section 106 Alternate Test Procedure

40 CFR 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", specifies approved test procedures for NPDES self-monitoring and data submitted to condition an NPDES permit.

Appendix H to 40 CFR 35, specifies approved test procedures be used by a pollution control agency to show compliance or non-compliance with an NPDES permit. Other monitoring programs (ex. - PCB toxic pollutant monitoring) specify the use of 40 CFR 136 test procedures.

40 CFR 136 selects specific documented test procedures from "Standard Methods", EPA's "Methods for Chemical Analysis of Water and Wastes", and "ASTM, Part 31", on a pollutant-by-pollutant basis, for the analysis of NPDES effluents. Based on the knowledge available to EMSL-Cincinnati, EPA, these test procedures were selected as the best available test procedures for effluent analysis - physical, chemical or microbiological.

40 CFR 136.4 and 136.5 specifies that alternate test procedures may be used if approval either is obtained from the Regional Administrtator on a case-by-case basis, or from the U.S. EPA Administrator on a nationwide basis. Alternate test procedures are justified by, but are not limited to, increased analytical performance and increased cost effectiveness to the approved method(s), or are proposed because they are promising new

methodologies. Alternate test procedure applications are processed by U.S. EPA if an applicant can justify their use for NPDES monitoring.

The Regional Administrator approves alternate test procedures on a case-by-case basis for specific NPDES permits within the specific U.S. EPA REgion and specific laboratories (public agency and commercial) receiving NPDES samples from a finite portion of an EPA Region. Appendix H to 40 CFR 35 specifically provides authority to the Regional Administrator for approval of alternate test procedures in State laboratories.

a. NPDES Alternate Test Procedures for Nationwide Use

Contact the Director, Environmental Monitoring and Support Laboratory (EMSL)-Cincinnati, EPA, Cincinnati, Ohio 45268, phone (513)684-7301 or phone FTS 684-7301 for the protocol concerning alternate test procedures for nationwide use.

b. Elements of an NPDES Alternate Test Procedure Application on a Case-by-Case Basis

Until and unless printed application forms are made available from the U.S. EPA, any person may apply to the Regional Administrator in the Region where the discharge(s) occurs, through the Director of the State Agency having authority to issue NPDES permits within such State.

An application should be made in triplicate to the Regional Administrator and shall:

- Provide the name, address, and telephone number of the responsible person, firm or public agency making application.
- Identify the pollutant(s) for which approval is sought.
- Specify the applicability of the proposed test procedure. Applicability of an alternate test procedure can be sought for (1) one or more specific NPDES permits (in this case, the applicable I.D. number(s) must be provided), (2) all or certain types of NPDES discharges monitored, within a geographical area of the Region, by a commercial laboratory or by a pollution control agency laboratory (State or Federal); or (3) all or certain types of non-point source monitoring provided by a pollution control agency laboratory as part of a Section 106 or 208 program.

o Provide a justification for use of the proposed method. This can be, but is not limited to, increased analytical performance or cost effectiveness.

o Provide a detailed description of the proposed alternate test procedure. This should be written in sufficient detail that another laboratory could reproduce the applicant's equipment and instrumentation. This is a necessary part of the application, since final approval can only be given for a documented test procedure description. Suggested formats for a detailed description can be found in "ASTM, Part 31", "Standard Methods", or EPA's "Methods for Chemical Analysis of Water and Wastes".

o Provide the concentration range of interest for the pollutant(s) identified in the above item. In the case of specific NPDES permits, present and expected effluent limitation concentrations shall be documented. In the case of non point source waters, the criteria or standards, which the monitoring program is to assess, shall be documented.

o Provide the detection limit, and its definition for the proposed alternate test procedure.

o Provide copies of, or cite reference to any published studies, if available, on the applicability of the alternate test procedure to the NPDES effluent types in question.

o Provide data, using sample aliquots of representative waste effluents (and untreated or raw wastes, if appropriate), showing the proposed method yields results comparable in equivalency and precision to the reference method, or one of the reference methods, specified by 40 CFR 136. The comparability data protocol listed in one of the following two items will be used.

o For an NPDES discharger, with one to four effluents of the same waste characteristic, provide comparability data by the following protocol. Select at least eight different effluent aliquots, collected over a representative time period, to provide varying concentration levels of the pollutant of interest. Determine or measure the pollutant of interest by the proposed test procedure and by a reference method. Specify the reference method used. Spike each of 8 aliquots, described above, with the pollutant of interest. Select spike concentrations so that the present spike recovery can be calculated from the amount of spike added. The chemical compound, selected to use as a spike material should assess the complete proposed test procedure or be representative of the chemical compounds or products of interest in the industrial or municipal process of interest. For example, organic nitrogen or phosphorus compounds should be selected as a spiking material for a Kjeldahl nitrogen or total phosphorus test procedure in order to include assessment of the digestion steps. Orthophosphate or ammonia compounds would only assess suitability of the final measurement step. After spiking of the waste aliquots, determine the pollutant concentrations by both the proposed method and by the reference method of choice. Calculate percent recovery on the basis of the amount of spike added. Specify the chemical compound used as a spike material.

If it is expected that the average percent recovery for the spike added will be between 95% and 105%, that the chemical compound selected for spiking is appropriate, and that significant concentrations of the pollutant of interest is present in the waste effluent aliquots, it will be unnecessary to analyze spiked samples by the reference method. If inadequate spike recovery by the proposed method is obtained, spike recovery by the reference method must be provided for comparative purposes.

If it is expected that there will be undetectable amounts of the pollutant present, either by the proposed method or by the reference method in unspiked samples, then equivalency data must be provided using both spiked and unspiked samples by the two test procedures.

Provide precision data for comparability of the two methods, either by analyzing the above eight effluent aliquots in duplicate by the two methods or by selecting a single waste aliquot of representative and detectable pollutant concentration and analyzing at least eight replicate values by each method.

Tabulate the above date to show equivalency of the proposed method with a reference method, comparability or adequacy of spike recoveries and comparability of the two method's precision and accuracy. Each effluent aliquot selected for comparability data should be uniquely identified and described as to appropriate NPDES Permit Number. All data collected during the comparability studies must be provided. Provision of comparability data can not be made on a selective basis.

o For an NPDES discharger laboratory, State laboratory, commercial laboratory, or U.S. EPA laboratory seeking approval for use of an alternate test procedure for a variety of NPDES permits, for all NPDES permits within a finite geographical area of EPA, Region V, or for all NPDES permits for specific industrial or municipal categories in a finite geographical area of EPA, Region V, comparability data will be provided by the following protocol.

Provide equivalency data, by both the proposed and reference methods, using 15 to 25 aliquots. The aliquots selected must be representative of the applicability specified above.

Provide spike recovery data, as appropriate, for the above 15 to 25 aliquots. Specify the chemical compound used as a spike material as discussed above.

Provide precision data for comparison purposes, either by analyzing the above 15 to 25 aliquots in duplicate by the two methods, or by providing eight or more replicate analyses of at least three or more of the 15 to 25 aliquots by the two methods.

Tabulate all of the above data for comparative purposes.

Until or unless printed application forms or a national U.S. EPA policy for a comparability data protocol is implemented, a case-by-case NPDES alternate test procedure application in Region V will contain the above items. It is impossible to specify a comparability data protocol that is applicable in all situations. Applicants seeking a case-by-case approval are encouraged to contact the appropriate State pollution control agency or the Quality Assurance Office, Region V, EPA, prior to initiation of comparability studies which do not fit the protocols provided in above items. Examples of this are proposed test procedures for suspended solids which do not allow spiking, pollutants whose test procedure defines the pollutant (BOD, oil and grease, suspended solids, fecal coliform, etc.), and alternative sample preservation techniques or holding times. A protocol for obtaining approval of an alternative preservation technique or holding time, for limited applicability, is described below.

Requests are often made to monitor a certain parameter or pollutant in lieu of a pollutant specified by a NPDES permit (ex. - to monitor chemical oxygen demand to show BOD permit compliance, after a correlation factor has been established). It is the policy of the Quality Assurance Office, Region V, not to process these requests as alternate test procedure application. They should be processed as a request to modify an NPDES permit and should be directed to the Enforcement Division, Region V.

c. Elements of an NPDES Alternative Sample Preservation or Holding Time Application on a Case-by-Case Basis

NPDES alternate test procedure applications are applicable to replacement of a preservation technique or to extending a holding time specified by a reference method cited by 40 CFR 136. Applications for such requests are made to the Regional Administrator in the Region where the discharge(s) occurs, through the Director of the State agency having authority to issue NPDES permits within such State.

An application should be made, in triplicate, to the Regional Administrator and shall:

o Provide the name, address and telephone number of the person, firm or public agency making the request.

o Identify the pollutant(s) for which approval is sought.

o Specify the applicability of the proposed test procedure - i.e., specific NPDES Permit Numbers.

o Provide present and future NPDES effluent limitations in concentration units.

- Provide a justification for use of the proposed preservation technique - ex. - cost effectiveness.

- Provide a detailed description of the proposed alternative preservation technique or holding time. This is quite necessary since final approval can only be given for a documented test procedure. Suggested formats for a detailed description can be found in, "ASTM, Part 31", EPA's "Methods for Chemical Analysis of Water and Wastes", and "Standard Methods".

- If available, cite references or provide copies of published studies showing the applicability of the proposed technique.

- Provide data, using sample aliquots or representative waste effluents (and untreated or raw waste, if appropriate), showing the proposed preservation technique or holding time yields results comparable in equivalency (not biased against the approved technique) and in precision to the approved preservation procedure.

- For a single NPDES permitted effluent, provide comparability data by the following protocol.

Select at least fifteen different effluent aliquots, collected over a representative time period, to provide varying concentration levels of the pollutant of interest.

Each aliquot of waste should be split into four separate sample bottles at time of collection.

Two aliquots are to be analyzed by an approved test procedure using the approved preservation technique. The remaining two aliquots are to be analyzed, by the same test procedure, using the proposed preservation technique or holding time. The test procedure is to be specified. If the proposed preservation technique uses an extended holding time, then the maximum holding time, specified in the proposed preservation techniques detailed test procedure description, should be used.

Pollutant values should be tabulated for each of the four waste aliquots along with the corresponding dates of sample collection, dates of analysis using the proposed technique, and dates of analysis using the approved preservation technique.

All analysis values determined should be reported. Data can only be discarded on the basis of quality control audit or control solution values showing a specific set of analyses to be out-of-control.

All analyses, using the two preservation techniques, should be performed in a single laboratory using a single analytical methodology.

d. Section 106 of Public Law 92-500 Alternate Test Procedure Program

If approval of a NPDES alternate test procedure is given to to a State laboratory, then approval will also be given for remaining non-point source measurements in the State's Section 106 monitoring program, if so requested. The NPDES alternate test procedure's working concentration range should be appropriate for the needs of the Section 106 program.

If a State requests approval of an alternate test procedure for non-NPDES monitoring, it may do so without providing complete comparability data so long as a documented test procedure description is provided, there are sufficient published studies provided to demonstrate its utility, and/or there are sufficient intralaboratory quality control data in existence to document its utility. The Regional Administrator shall determine the need for additional comparability data upon the recommendations of the Quality Assurance Office, Region V.

8.3.2.2 Elements of an Application for a Safe Drinking Water Act (SDWA) Alternate Test Procedure

The National Interim Primary Drinking Water Regulations (NIPDWR), 40 CFR 141, implementing the SDWA, specifies test procedures to use for NIPDWR contaminants. 40 CFR 141.27 states that with the approval, both of a primacy State and of the EPA Administrator, a laboratory may use alternate test procedures. This authority has been delegated to the Regional Administrator.

A memorandum of March 10, 1977, from the Ofice of Water Supply (OWS), EPA, specifies the mechanisms for obtaining approval of SDWA alternate test procedures. Final approval is either given by the Regional Administrator on a case-by-case basis to specific water utilities, and State, Regional EPA, and commercial laboratories or by the OWS on a nationwide basis. The alternate test procedures for nationwide use should be published in the Federal Register.

The mechanism specified by the March 10th memo is extremly cumbersome, but it does designate who has authority for final decision making.

The Regional Administrator is responsible for final determination of alternate test procedures for approved water utility, State, commercial, and Regional EPA laboratories. EMSL-Cincinnati and OWS, both of EPA, are responsible for the determination of alternate test procedures for nationwide use. Alternate test procedures approved, as of September 1978, for nationwide use, are contained in two OWS memorandums of September 1, 1977, and March 9, 1978 (Appendix 15)

If approval of an alternate test procedure has been given by the Regional Administrator on a case-by-case basis, to a private or public laboratory for NPDES monitoring, approval for monitoring of the same pollutant as a SDWA contaminant will also be given by the Regional Administrator, upon request, so long as the original NPDES application clearly documents the alternate test procedure's working concentration range is applicable to measurement at the Maximum Contaminant Level (MCL) specified by the NIPDWR.

a. SDWA Alternate Test Procedure for Nationwide Use

Contact the Director, Environmental Monitoring and Support Laboratory (EMSL)-Cincinnati, EPA, Cincinnati, Ohio 45268, phone (513)684-7301 or phone FTS 684-7301, concerning the protocol for SDWA alternate test procedures for nationwide use.

b. Elements of a SDWA Alternate Test Procedure Application on a Case-by-Case Basis

Approval of an alternate test procedure can be requested by a water utility, public, or private laboratory that has made application for, or has, Interim Laboratory Certification under an existing State or Federal SDWA laboratory approval program in Region V, EPA.

Application for use of an alternate test procedure, on a case-by-case basis, is made in quadruplicate to the Regional Administrator, through the State water supply program for those State which have accepted primacy for the SDWA. In non-primacy States, application is made directly to the Regional Administrator.

Until or unless printed application forms are made available from the U.S. EPA on a national basis, a case-by-case application in Region V, EPA shall:

- Provide the name, address and telephone number of the responsible person making application.

- Identify the SDWA contaminant(s) for which approval is sought.

- Specify the applicability of the proposed test procedure either for a specific utility, or utilities, or for a specific public agency or commerical laboratory doing work for utilities.

- Provide a justification for use of the proposed methodology instead of a reference methodology.

- Provide a detailed description of the proposed test procedure. See "ASTM, Part 31", EPA's "Methods for Chemical Analysis of Water and Wastes", or "Standard Methods", for suggested formats.

- Provide data showing the proposed method yields results comparable in equivalency and precision to a reference method or an alternate test procedure approved for nationwide use, in the concentration range of the NIPDWR MCL. Comparability data for 1 to 4 utilities, of equivalent water characteristics, shall be provided using the NPDES protocol for eight effluent aliquots. If approval is sought by a commercial or public agency laboratory for use for all utilities in a State, then the NPDES comparability data protocol using 15 to 25 different water utility aliquots should be utilized. Sample aliquots will have to be spiked, at or near the contaminant's MCL, and should be measured by both the proposed and approved methods. Organo arsenic and organo mercury compounds shall be used as spiking compounds for these two contaminants, because their reference methods contain specific digestion procedures. For a wide applicability, sample aliquots, selected for comparability measurements must be a wide cross-section of the potable water in a State.

- Provide the proposed method's detection limit and precision at the contaminant's MCL. The terms detection limit and precision shall be defined by the applicant.

8.3.2.3 Processing of Case-by-Case Alternate Test Procedure in Region V

a. A NPDES discharger, water utility, or laboratory should make application in triplicate, for use of a case-by-case alternate test procedure, to the Regional Administrator, through the responsible State authorities having authority to enforce the National Pollutant Discharge Elimination System (NPDES) program or the Safe Drinking Water Act (SDWA). An extra application copy is provided State authorities. If the State does not have appropriate enforcement authority, then application is made, in triplicate, directly to the Regional Administrator.

b. Application for nationwide use of an NPDES or SDWA alternate test procedure is made directly to the Director, EMSL-Cincinnati, EPA, Cincinnati, Ohio 45268, in accordance with EMSL-Cincinnati's protocols.

c. For a case-by-case application, the State authorities will forward three copies of the application to the Regional Administrator with the States' recommendations. The regulations specify the State agency Director shall forward this application to the Regional Administrator. Guidance from the Office of Water Supply (OWS), EPA, specifies this shall be done by an appropriate State Official.

d. Upon receipt of the application with State recommendations, (when the application is applicable to a State with delegated enforcement responsibility), the Regional Administrator will forward the application, in triplicate, to the Quality Assurance Office (QAO), Region V, for processing. Upon receipt of the application, the QAO, Region V, will acknowledge receipt of the application to the applicant. This starts the time cycle for action on the request so that a final determination on the request can be made within 90 days by the Regional Administrator.

e. If a State with enforcement responsibility for the SDWA or NPDES program recommends disapproval of the proposed test procedure, the Regional Administrator shall deny the application. Copies of this disapproval will be sent to the appropriate State agency and to its State Laboratory Director, to the Director, EMSL-Cincinnati, and in the case of SDWA applications to the Office of Water Supply (OWS), EPA.

f. The QAO will review the application for the following:

- o A clear understanding of the applicability of the proposed test procedure.
- o A test procedure documented in sufficient detail that another laboratory could reproduce the results of the applicant's laboratory.
- o Comparability data in sufficient quantity and consistency with the proposed applicability of the alternate test procedure.
- o Consistency with the data quality needs of the SDWA, NPDES program, and other monitoring programs as appropriate.

If any of the four elements are missing, the QAO will request the necessary information from the applicant within one month of receipt of the application. When the applicant provides this information to the QAO, a new 90 day cycle will be initiated.

g. If the application is complete, the QAO will forward a copy to the Director, EMSL-Cincinnati for his technical review and recommendations, within two weeks of receipt of the alternate test procedure application.

h. At the discretion of the QAO, a copy of the application will be forwarded to the Water Supply Branch, Region V, or to the Enforcement Division, Region V, for their recommendations, if program policies are affected by either approval or disapproval of the application.

i. Within the 90 day time period, the QAO will receive all appropriate recommendations and prepare a letter for the Regional Administrator's signature to notify the applicant of approval of rejection, and in some instances specify the additional information which is required to deteremine whether to approve the proposed test procedure. Copies of this final determination by the Regional Administrtor shall be forwarded to appropriate Regional and State program personnel, to the State Laboratory Director(s) of the concerned State(s), to the Director, EMSL-Cincinnati, and in the case of SDWA approvals, to the Director, OWS. The QAO will prepare the final determination letter for the Regional Administrator so that this determination reflects final authority by the Region and is consistent with broad national EPA policies.

8.3.2.4 Procedures for Equivalent Test Prodcedure Under the Clean Air Act

Methods required by the Clean Air Act are designated as Reference of Equivalent Methods according to 40 CFR 53.1. The use of methods which are not so designated must be approved by Department E, EMSL-RTP. Procedures for obtaining approval of a non-designated method are described in 40 CFR 53.4 and 40 CFR 53.14, respectively. These procedures require that any user modifications which are not reference or equivalence must be approved by Department E, EMSL-RTP.

8.4 Instrumentation

All monitoring equipment and instrumentation pruchased within Region V with EPA grant, contract, inter-agency agreement, or operation funds are to be evaluated and recommended for approval or rejection by the QAO. For external monitoring projects, including 201 grants used to purchase monitoring equipment, the Project Officer will submit the equipment or instrumentation request and any justifications to the QAO through appropriate channels for review. Internal office Directors and Branch Chiefs (Region V) will submit their proposals and justifications through the appropriate Division/ Office Director to the QAO through the Surveillance and Analysis Director for review. As part of the evaluation and approval process the following minimum points are considered.

- o Is there a need, present or future for the item, i.e., does present or projected regulations specify tests that this equipment will be used for.
- o Does the purchaser have equipment in-house that can be modified or adapted to perform the necessary function at a lesser cost.
- o Will the purchaser have the necessary auxillary in-put, eg., if G.C. - Mass spectroscopy unit is requested, will library facilities be available.
- o Are there technically competent personnel available to operate the equipment. If not, what plans are available for hiring or training such personnel.

The QAO will forward an official recommendation of approval for funding/purchase or a recommendation for not funding/purchase to the appropriate project officer, Division/Office Directory through appropriate channels. In the case of not recommending funding/puchase a justification is also provided.

8.5 Calibration and Standards

Calibration procedures require the application of primary or secondary standards. The standards used, whether they are apparatus or reagent standards are to be certified as being traceable to standards of the National Bureau of Standards, or other recognized fundamental standard. This type of traceability is possible when standards are generated in the laboratory. Regardless of the type of calibration equipment or material, an effective QA program requires accuracy levels of these materials that are consistent with the method of analysis. The calibration policies and procedures outlined in 8.5 apply to all measuring and test equipment/instrument associated with a monitoring activity, including:

- Sampling equipment at sampling stations
- Analytical equipment/instruments in the laboratory
- Flow measuring devices (eg., current meters, rotameters), volume (eg., dry gas meters), pressure, vacuum and temperature measurement equipment at the sampling station and in the laboratory.

As part of a monitoring activity's (Federal, State, local agency, contractor or grantee) QA program a written step-by-step procedure for a frequency for calibration of measuring and test equipment/instruments and use of calibration standards is to be provided, in order to eliminate possible measurement inaccuracies due to difference in techniques, environmental conditions, choice of higher level standards and compliance with Agency regulations (eg., 40 CFR Part 58, Appendix A and B). As a minimum, these procedures are to include the following:

1. The specific equipment or group of equipment (instruments) to which the procedure is applicatable. Equipment of the same type, having compatable calibration points, environmental conditions, and accuracy requirements, may be serviced by the same calibration procedure.

2. A brief description of the scope, principle, and/or theory of the calibration method.

3. Fundamental calibration specification, such as calibration points, environmental requirements, and accuracy requirements.

4. A list of calibration standards and accessory equipment required to peform an effective calibration. Manufacturer's name, model number, and accuracy should be included as applicable.

5. A complete procedure for calibration arranged in a step-by-step manner, clearly and concisely written.

6. Calibration procedures are to provide specific instructions for obtaining and recording the test data, and include data sheets that are to be used.

7. A detailed documented sample of computations for any calibration procedure that requires statistical analysis of results.

8. All field and laboratory calibration are to be traceable through an unbroken chain (supported by reports or data sheets) to some ultimate or national reference standard.

9. An up-to date- report for each calibration standrd used in the calibration system is to be made available for review during the QAO's audit or on-site system evaluation of any monitoring activity in Region V, funded by EPA.

All equipment past due for calibration should be removed from service either physically or, if this is impractical, should be impounded by tagging or other means.

The monitoring activity's quality control official or other individual delegated quality control responsibility (e.g., Laboratory Section Chief) has day-to-day responsibility to ensure that the monitoring activity maintains the required accuracy in the calibration program.

The QAO will evaluate the monitoring activity's on going calibration and standards activity as part of the audit and on-site evaluation process to ensure valid data is being produced. Problems will be identified and recommendations for corrective action provided. The QAO cannot validate data that is suspect. Follow-up to validate and approve correction actions will be QAO's responsibility.

8.6 Preventive Maintenance and Inspections

As defined here, preventive maintenance is an orderly program of positive actions (equipment cleaning, lubricating, reconditioning, adjustment and/or testing) for preventing failure of monitoring systems or parts thereof during use. The most important effect a good preventive maintenance program has is to increase measurement system reliability and thus increase data completeness. Conversely, a poor preventive maintenance program will result in increased measurement system downtime (i.e., decrease in data completeness) and in increased unscheduled maintenance costs; and may cause distrust in the validity of the data. In ambient air monitoring, data completeness criteria are used to validate data.

A responsible individual (i.e., field section Chief, laboratory Section Chief, QC Officer) is required to prepare and implement a preventive maintenance schedule for all equipment and measuring systems, as part of the monitoring activity's total QC program. The planning required to prepare the preventive maintenance schedule will have the effect of:

1. Highlighting that equipment or those parts therof that are most likely to fail without proper preventive maintenance.

2. Defining a spare parts inventory that should be maintained to replace worn-out parts with a minimum of downtime.

A specific preventive maintenance schedule is to relate to the purpose of testing, environmental influences, physical location of equipment, and the level of analyst skills. Checklists are to be used as documentation for listing specific maintenance tasks and frequency (time interval between maintenance). In some instances, if calibration tasks are difficult to separate from preventive maintenance tasks, a combined preventive maintenance - calibration schedule is advisable.

A record of all preventive maintenance and daily service checks are to be maintained. An acceptable practice to follow for recording completion of task is to maintain a preventive maintenance calibration multiple copy log book. After tasks have been completed and entered in the log book, a replicate copy of each task is removed by the individual performing the maintenance - calibration task and forwarded to the appropriate supervisor and QC Officer for review and conformance with monitoring activity's preventive maintenance protocol. The log book is stored by the instrument for future reference. The QAO will review these log books during the audit or on-site systems evaluation activity for deficiencies.

8.7 Quality Control Procedures

Assuming that all basic variables pertaining to laboratory services (i.e., instrumentation, glassware, reagents, solvents, gases, etc.) are under control, that approved methods are being used, and the complete system is initially under quality control, valid precision and accuracy data must initially be developed for each method and analyst. Then, to insure that valid data continue to be produced, systematic daily checks must show that the test results remain reproducible, and that the methodology is actually measuring the quantity in each sample. In addition, quality control must begin with sample collection and must not end until the resulting data have been reported. Quality control of analytical performance within the laboratory is thus but one vital link in generation and dissemination of valid data for agency use. Understanding and conscientious use of quality control among all field sampling personnel, analytical personnel, and management personnel is imperative. Region V's procedures are outlined in the following Sections (8.7.1 and 8.7.2). Management of QC procedures (how and by whom) is described in Section 10.2.

8.7.1 Intra-Laboratory Quality Control Procedures

The purpose of intralaboratory QC programs is to identify the sources of measurement error and to estimate their bias (accuracy) and variability (repeatability and replicability). For manual measurement methods, bias and variability are determined separately for sample collection and analysis and are combined for determination of total method bias and variability. For continuous methods, total method bias and variability are determined directly. Some of the potential error sources are the operator or analyst, equipment, the calibration, and the operating conditions. The results may be analyzed by making comparisons against each other and/or against reference standards. To maintain a known level of competence in daily activities, quality control must be implemented in the field and at the bench, using a system of checks to determine the accuracy and precision of results and the performance of measurement systems and operators. Intralaboratory quality control is a continuing activity to insure the output of data of known quality. The specific objectives are to devise a program that:

o measures and control the precision of procedures and instruments.

o measures and control the accuracy of analytical results.

- documents, on a continuous basis, the performance of systems analysts and operators.
- establishes training needs.
- identifies weak measurement methodology and provides feedback to the Quality Assurance Office, where an evaluation can be made of the findings and the appropriate EMSL group notified so method revisions and/or modifications can be made.

In Region V quality control charts are to be the foundation of the laboratory's interlaboratory quality control programs. One form of quality control showing trends is the summation of the differences squared for replicate samples. Additional control charts are recommended where standard deviations are $(d = Vd^2/k)$ for use on a daily basis to establish rapidly if an analysis is out of control on a given day. Once precision and accuracy data are available on the method and the operator/analyst, systematic daily checks are necessary to ensure that valid data are being generated. From these daily precision and accuracy data, quality control charts can be constructed and maintained to determine when the method used is producing valid data, when the data are questionable, or when a trend is detected which must be investigated and corrected.

Several techniques are available for constructing quality control charts and plotting subsequent data. The two techniques currently used by EPA are the Shewhart technique and/or Cumulative-Summation technique. These techniques are depicted in (EPA Publications) EPA-600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, March 1979, and Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I (EPA-600/9-005), Volume II (EPA-600/4-77-0272a) and Volume III (EPA-660/4-77-027b). For both techniques, precision control charts are constructed from duplicate sample analyses, and accuracy control charts are constructed from spiked sample analysis, utilizing standard reference materials (SRM). SRM's are substances which qualify as absolute quantities against which other like substances can be calibrated or measured. The SRM, typically produced by organizations like the National Bureau of Standards (NBS), is used to prepare standard reference standards (SRS) for routine laboratory and field use.

SRS (also referred to as spiked samples) are preparations of known amounts of standard reference materials added to an actual environmental samples which has been previously analysed. The amount of the substance found in the sample is a "true" indication of the accuracy of the method for a given measurement. The use of the standard reference samples measures the extent of interferences which cannot be obviated.

Following normal procedures, the control chart must indicate the conditions under which it was developed; i.e., laboratory name, parameter, method of analysis, date of preparation, and any other information unique to the initializing data such as range of concentration and identification of analyst(s)/operator. A control chart is not generally applicable under other conditions.

To verify the accuracy and precision of control charts, the initializing data should be checked to be sure that none of the values exceeds these new control limits. In addition, if its distribution is proper, about 68 percent of the initializing data should fall within the interval average percent recovery plus or minus 2 times the standard deviation for percent recovery. There is a question of validity of the control chart if less than 50% of the initializing data falls within this interval.

In application of the accuracy control chart, either of the following two conditions indicates an out-of-control situation.

a. Any point beyond the control limits.

b. Seven successive points on the same side of the interval average percent recovery of the central line of the completed control chart.

When an out-of-control situation occurs, analyses shall be stopped until the problem has been identified and resolved, after which the frequency should be increased for the next few percent recovery QC checks. The problem and its solution must be documented, and all analyses since the last in-control point must be repeated or discarded.

For some parameters it may be necessary to construct low level and moderate to high level accuracy QC charts for each standardization concentration level sample.

In application of the precision control chart, the chart should be updated periodically as additional, or more current, data become available, or whenever the basic analytical system undergoes a major change. If any difference between duplicate analyses exceeds the critical-range value for the appropriate concentration level, then analyses should be stopped until the problem is identified and resolved, and the frequency is to be increased for the next few precision checks. After resolution, the problem and its solution must be documented, and all analyses since the last in-control check must be repeated or discarded.

Once the quality control charts have been developed and put in place, the normal day to day working routine requires the following:

- A new standard curve should be established with each new batch of reagents, using at least seven concentration levels. The number of level in continuous monitors is 3 levels within range.
- With each batch of analyses (10 to 20% of the time), the following tests are to be run:
  1. One blank on water and reagents.
  2. One midpoint standard.
  3. One standard reference standard (spike) to determine recovery.
  4. One set of duplicate analysis.

The results from 2 through 4 are to be compared with previous in-control data by using the protocol specified above (for a detailed protocol description refer to Section 6.3 of Chapter 6 of EPA Publication 600/4-79-019).

The following protocol is to be implemented to indisputably establish the validity of data for each parameter from water and wastewater projects:

In the following protocol the symbols used represent the results of analysis according to the scheme:

A_1 = first replicate of sample A

A_2 = second replicate of sample A

B = sample taken simultaneously with sample A

B_{SF} = field spike into sample B

B_{SL} = laboratory spike into sample B

D_F = field spike into distilled water

D_L = labortory spike into distilled water

T = true value for all spikes

The laboratory spikes B_{SL} and D_L are the only analyses that may not be necessary. All other analyses must be done simultaneously.

Field personnel should perform the following steps for quality assurance.

a. Take independent simultaneous samples A and B at the same sampling point. Depending on the parameter, this might involve side-by-side grab samples or composite samplers mounted in parallel.

b. Split sample A into the equal-volume samples A_1 and A_2.

c. Split sample B into equal volumes and add a spike T to one of them; the latter sample becomes sample B_{SF}. As with all spikes, the addition of T should approximately double the anticipated concentration level.

d. Add the same spike T to a distilled water sample furnished by the alboratory and designate this sample as D_F.

These QC samples must be treated in the same way as routine samples; i.e., the volume, type of container, preservation, labeling, and transportation must be same for all.

The laboratory pesonnel should perform the following steps for quality assurance:

a. Analyze the blank and midpoint standard recommended in the normal day-to-day working routine. If results are unsatisfactory, resolve problems before continuing.

b. Analyze sample D_F. If the percent recovery of T is unsatisfactory (see accuracy protocol), create a similarly spiked, distilled-water sample D_L and analyze to test for a systematic error in the laboratory or fundamental problems with the spike. If the percent recovery of T from D_L is satisfactory, any systematic error occurred before the samples reached the laboratory.

c. Analyze samples B and B_{SF}. If B is below the detection limit, or if B is greater than 10T or less and 0.1T, disregard the remainder of this step and proceed to step d. If the percent recovery of T from B_{SF} is unsatisfactory (see accuracy protocol), spike an aliquot of sample B the same way in the laboratory so that a similar recovery can be anticipated. Analyse this sample B_{SL} to test for immediate sample interferences or a bad background result B. If the percent recovery from B_{SL} is satisfactory, then the interference must require a longer delay before analyses, or other special conditions not present in the laboratory, in order to have a noticeable effect upon recovery of the spike.

d. Analyze A_1 and A_2. If the absolute (unsigned) difference between these results exceeds the critical value (see precision protocol), then test of precision is out of control.

e. Calculate the absolute difference between A_1 and B. If it is unsatisfactory (see precision protocol), the field sampling procedure did not provide representative samples.

If initial results at each of the laboratory steps were satisfactory, then the validity of the related data has been indisputably established. If results at any step are unsatisfactory, resolution depends upon the problem identified. Laboratory problems may just require that the analyses be repeated, but field problems will usually require new samples. Figure 8.7.1 is intended to clarify the interdependence of the preceeding laboratory steps b through e.

In figure 8.7.1 it must be noted that there is no way to identify additive sample interferences; i.e., those that have an equal effect upon the background-plus-spike results (B_{SF} or B_{SL}) and the background result B. Recovery of a spike will not show such interferences.

Problems causing systematic errors that may occur in the field include the following:

a. Contaminated preservative, distilled water, or containers

b. Contamination by sampling personnel

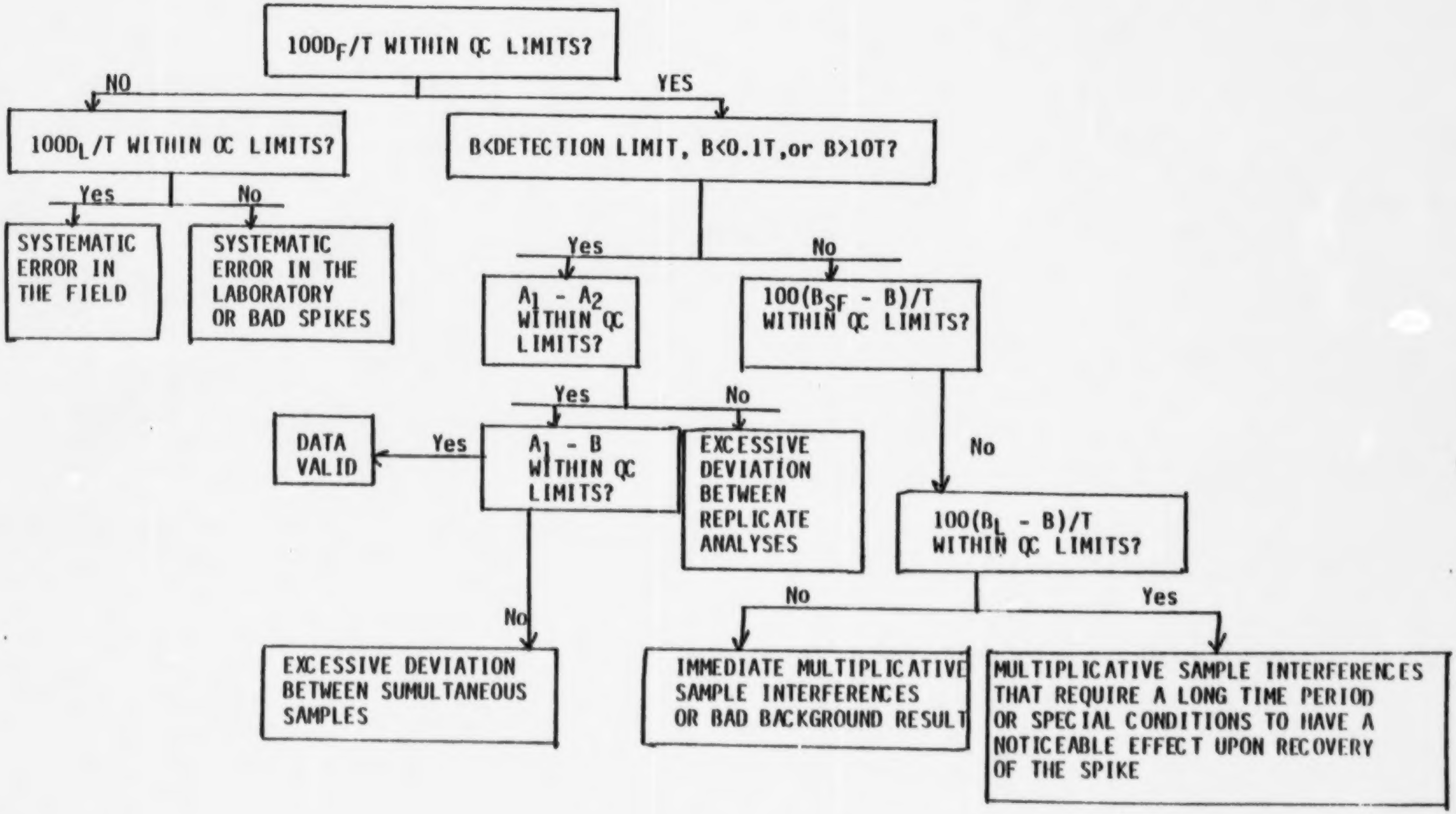
$100D_F/T$ WITHIN QC LIMITS?
NO
YES
$100D_L/T$ WITHIN QC LIMITS?
B<DETECTION LIMIT, B<0.1T,or B>10T?
Yes
No
SYSTEMATIC ERROR IN THE FIELD
SYSTEMATIC ERROR IN THE LABORATORY OR BAD SPIKES
Yes
No
$A_1 - A_2$ WITHIN QC LIMITS?
$100(B_{SF} - B)/T$ WITHIN QC LIMITS?
Yes
No
DATA VALID
Yes
$A_1 - B$ WITHIN QC LIMITS?
EXCESSIVE DEVIATION BETWEEN REPLICATE ANALYSES
No
$100(B_L - B)/T$ WITHIN QC LIMITS?
No
No
Yes
EXCESSIVE DEVIATION BETWEEN SUMULTANEOUS SAMPLES
IMMEDIATE MULTIPLICATIVE SAMPLE INTERFERENCES OR BAD BACKGROUND RESULT
MULTIPLICATIVE SAMPLE INTERFERENCES THAT REQUIRE A LONG TIME PERIOD OR SPECIAL CONDITIONS TO HAVE A NOTICEABLE EFFECT UPON RECOVERY OF THE SPIKE

Figure 8.7.1 Procedure for evaluating QC data from a monitoring study.

c. Deterioration through excessive holding time or use of an ineffectual preservation technique

d. Use of a bad field spiking procedure

8.7.1.2 Intra-Field Quality Control Procedures

Quality control programs for sampling equipment and for field measurement procedures (of such parameters as temperature, dissolved oxygen, pH and conductance) are necessary to insure data of the highest quality. A field quality control program administered by a quality assurance coordinator should contain the following documented elements:

a. The analytical methodology; the special sample handling procedures; and the precision, accuracy, and detection limits of all analytical methods used.

b. The basis for selection of analytical and sampling methodology. For example, all analytical methodology for NPDES permits shall be that specified by the Agency or shall consist of approved alternative test procedures. Where methodology does not exist, the quality assurance plan should state how the new method will be documented, justified, and approved for use.

c. The amount of analyses for quality control expressed as a percentage of overall analyses, to assess the validity of data. The complete quality control program is to specify 5% as a minimum for time assigned to field QC. The plan should include a shifting of these allocations or a decrease in the allocations depending upon the degree of confidence established for collected data.

d. Procedures for the recording, processing, and reporting of data; procedures for review of data and invalidation of data based upon QC results.

e. Procedures for calibration and maintenance of field instruments and automatic samplers.

f. A performance evaluation system, administered through the quality assurance coordinator, allowing field sampling personnel to cover the following areas:

   (1) Qualifications of field personnel for a particular sampling situation.

(2) Determination of the best representative sampling site.

(3) Sampling technique including location of the points of sampling within the body of water, the choice of grab or composite sampling, the type of automatic sampler, special handling procedures, sample preservation, and sample identification.

(4) Flow measurement, where applicable.

(5) Completeness of data, data recording, processing, and reporting.

(6) Calibration and maintenance of field instruments and equipment.

(7) The use of QC samples such as duplicate, split, or spiked samples to assess the validity of data.

g. Training of all personnel involved in any function affecting the data quality.

Quality assurance in sample collection is to be implemented to minimize such common errors as improper sampling methodology, poor sample preservation, and lack of adequate mixing during compositing and testing.

At selected stations, on a random time frame, duplicate samples are collected from two sets of field equipment installed at the site, or duplicate grab samples are collected. This provides a check of sampling equipment and technique for precision.

A representative subsample from the collected sample is removed and both are analyzed for the pollutants of interest. The samples may be reanalyzed by the same laboratory or analyzed by two different laboratories for a check of the analytical procedures.

Known amounts of a particular constituent are added in the field to an actual sample or to blanks of deionized water at concentrations at which the accuracy of the test method is satisfactory. The amount added and frequency is coordinated with the laboratory. This method provides a proficiency check for accuracy of the analytical procedures.

Acids and chemical preservatives can become contaminated after a period of use in the field. The sampler should add the same quantity of preservative to some distilled water as normally would be added to a wastewater sample. This preservative blank is sent to the laboratory for analysis of the same parameters that are measured in the sample and values for the blank are then subtracted from the sample values. Liquid chemical preservatives should be changed every 2 weeks, or sooner, if contamination increases above predetermined levels.

A minimum of seven sets each of comparative data for duplicates, spikes, split samples, and blanks should be collected to define acceptable estimates of precision and accuracy criteria for data validation of field parameters.

Protocol is to be developed and implemented for calibrating all field analysis test equipment and calibration standards to include the following: (a) calibration and maintenance intervals, (b) listing of required calibration standards, (c) environmental conditions requiring calibration, and (d) a documented record system. Written calibration procedures should be documented and should include mention of the following:

a. To what tests the procedure is applicable.

b. A brief description of the calibration procedure.

c. A listing of the calibration standard, the reagents, and any accessory equipment required.

d. Provisions for indicating that the field equipment is labeled and contains the calibration expiration date.

8.7.1.3 Additional Intralaboratory Quality Control Procedures for Specific Groups of Parameters

a. Microbiology intralaboratory quality control.
A quality assurance program for microbiological analyses must emphasize the control of laboratory operations and analytical procedures because the tests measure living organisms that continually change in response to their environment. Further, because true values cannot be provided for the microbial parameters, microbiologists do not yet have the advantages of analytical standards, QC charts, and spiked samples available to other disciplies for measurement of

accuracy. Because known values cannot be applied, it is important that careful and continuous control be exerted over sampling, personnel, analytical methodology, materials, supplies, and equipment.

A documented interlaboratory QC is to address the following areas as a minimum:

o An Operating Manual shall be prepared which describes the sampling techniques, analytical methods, laboratory operations, maintenance and quality control procedures. Specific details are given on all procedures and quality control checks made on materials, supplies, equipment, instrumentation and facilities. The frequency of the checks, the person responsible for each check (with necessary back-up assignments), the review mechanism in the QC program to be followed, the frequency of the review and the corrective actions to be taken are specified. A copy is provided to each analyst.

Part V of EPA Publication EPA-600/8-78-017 describes the normal day-to-day microbiology interlaboratory QC routine, to be implemented by Region V microbiology laboratories. A record is to be maintained of the daily QC checks and procedures. If there is no proof of performance, and evidence for future reference, for practical purposes, no QC program is in operation.

o A Sample Log shall be maintained which records, chronologically, information on sample identification and origin, the necessary chain of custody information, and analyses performed.

o A Written Record shall also be maintained of all analytical QC checks: positive and negative culture controls, sterility checks, replicate analyses by an analyst, comparative data between analysts, use-test results of media, membrane filters and laboratory pure water, replicate analyses done to establish precision of analysts, or of methodology used to determine non-compliance with bacterial limits established by Agency regulations.

b. Aquatic Biology Interlaboratory Quality Control

Interlaboratory QC procedures for aquatic biology programs are fully described in EPA Publication, EPA-679/4-73-001.

An operating manual shall be prepared, addressing the following essential elements as a minimum:

1. An understanding and acceptance of the importance of quality control (QC) and a commitment on the part of the biology staff to fully integrate QC practices into field and laboratory operations.

2. Staff needs with adequate formal training and experience and proper specialization to meet program needs.

3. Adequate field equipment, storage and laboratory space, instrumentation, and taxonomic references.

4. Protocol for preparation and design of field and laboratory studies.

5. Documentation of approved methodology, where available, and protocol for consideration of the tehcnical defensibility of the methods and their application.

6. Protocol for use of replication in sample collection and analyses where feasible, and determination of the accuracy and precision of the data.

7. Protocol for frequent calibration of field and laboratory instruments.

8. Protocol for proper sample identification and handling to prevent misidentification or intermixing of samples.

9. Protocol for blind, split, or other control samples to evaluate performance.

10. Procedures for development and regular use of in-house reference specimen collections, and use of outside taxonomic experts to confirm or provide identifications for problem specimens.

11. Procedures for meticulous, dual-level review of the results of manual arithmetical data manipulations and transcriptions before the data are used in reports or placed in BIO-STORET.

c. Organic Chemistry Interlaboratory Quality Control

Most of the quality control program described above in Section 8.7 of this document cannot easily be adapted to the methods for organic compounds. Therefore, the Agency has developed a series of tests and protocols whose purpose is to describe the performance of the computerized gas chromatography - mass spectrometry systems and the analysts(s). The complete protocol procedure is listed in Appendix 16. A summary of these performance tests follows:

I. Spectrum Validation Test - Uses decafluorotriphenyl phosphine (DFTPP) to deteremine whether the system gives a 70 ev electron ionization fragmentation pattern similar to that found in the historical mass spectrometry data base, and the required mass resolution and natural abundance isotope patterns. The spectrum of DFTPP must meet the criteria given in Table 2 of Appendix 16.

II. System Stability Test - Uses DFTPP to test moderate term (20-28 hours) system stability. The criteria given in Test I must be met.

III. Instrument Detection Limit Test - Uses DFTPP to measure the full and valid spectrum detection limit at a defined and tolerable noise level. At a signal/noise = 5, the required instrument detection limits are 50 nanograms for systems used in the anlaysis of industrial or municipal wastes, and 30 nanograms for systems used in the analysis for ambient or drinking water.

IV. Saturation Recovery Test - Uses DFTPP and p-bromobiphenyl to simulate a frequently encountered situation with real samples. The spectrum of DFTPP, measured within two minutes after the elution of a 250 fold excess of p-bromobiphenyl, must not contain significant contributions from the ions attributable to p-bromobiphenyl.

V. Precision Test - Uses a variety of typical environmental pollutants to determine precision from filling a syringe to peak integration. The mean relative standard deviation for the compounds used in the test which elute as narrow peaks must be 7% or less using either peak areas in arbitrary units or ratios of peak areas. For broad peaks the mean relative standard deviation must be 13% or less.

VI. Library Search Test - Uses data from Test V to evaluate the speed and completeness of the minicomputer library search algorithm. The mean search time, including background subtraction, must be one minute or less, and all test compounds must be identified as most probable except isomers with very similar spectra should not be counted as incorrect.

VII. Quantitative Analysis with Liquid-Liquid Extraction - Uses a variety of environmental pollutants to measure quantitative accuracy and precision of the total analytical method. The mean of the means of the percentages of the true values observed must be in the 68-132% range with a mean relative standard deviation of 38% or less using either internal or external standards. This test also evaluates laboratory performance.

VIII. Quantitative Analysis with Inert Gas Purge and Trap - Uses a variety of compounds to measure quantitative accuracy and precision of the total analytical method. The mean of the mean method efficiencies must be 70% or more. Chloroform efficiency must exceed 90% and all compounds must exceed 30% efficiency. The spectrum of p-bromofluorobenzene must meet the criteria given in Table 7, Appendix 16. The mean of the means of the percentages of the true values observed must be in the range of 90-110% with a mean relative standard deviation of 19% or less using either internal or external standards.

IX. Qualitative Analysis with Real Samples - Uses a real sample to evaluate the ability of the system to deal with real sample matrix effects and interferences. All compounds must be correctly identified except isomers with nearly identical mass spectra should not be counted as incorrect. This test also evaluates laboratory performance.

X. Solid Probe Inlet System Test (Optional) - Uses cholesterol to evaluate the spectrum validity achievable with a solid probe inlet system. The spectrum of cholesterol must meet the criteria given in step three of the test.

The performance tests are intended for use in the evaluation of the system initially and on a long term basis. All tests are to be initially performed with correction being made to meet the criteria established in Appendix 16 for the respective test. The results and corrections are to be documented.

Test I performance is to be revalidated on a daily basis. Test IV, V, VII, VIII, and IX performance are to be revalidated on a specified frequency identified in the documented intralaboratory QC protocol.

The normal day-to-day QC routine is divided into three separate categories. Data generated from each category is documented. Problems identified must be corrected and documented. When out of control situations occur, analyses shall be stopped until the problem has been identified and resolved.

The first category represents the determination of purgeable compounds. This determination is performed in a closed analytical system; the complete analysis can be performed in 1 h; and the number of theoretically possible interferences is somewhat limited. The second category represents liquid/liquid partition methods in a regulatory situation. Here a very limited number of compounds are being measured; there is a high occurrence of positive results; and it is important to establish that the method works satisfactorily on the particular sample matrix. The third category represents liquid/liquid partition methods in a monitoring situation. Here a large number of compounds are often being measured simultaneously; there is a low occurrence of positive results; and each sample matrix may be different. Quality assurance is aimed at establishing that the laboratory is using the method correctly.

The purgeable methods are unique among organic methods because the standards are treated in exactly the same way as the samples, and there is no inherent method bias. The methods are amendable to a variety of quality assurance programs. The approach that has been found applicable to all types of samples and provides the maximum data for the expended effort consists of the addition of one or more internal standards to the matrix before purging. Data generated in this program provide a continuous monitoring of the equipment and establishes matrix applicability for the test.

For liquid/liquid extraction methods in a regulatory situation, the emphasis is placed on duplicates and dosed samples. Both field duplicates and laboratory duplicates are used in the program to establish sampling and subsampling validity.

The dosing of samples to establish method accuracy for the matrix is an integral part of this program. Where the analytical program will extend over a long period of time the construction of control charts is recommended.

When the liquid/liquid extraction methods are used for monitoring, the emphasis is placed on an external control series. A standard laboratory matrix is developed. With each series of samples the matrix is dosed and analyzed with the samples. Data generated over a period of time can be used to monitor the performance of the equipment and the analyst, with relatively tight specifications to define problems that arise. Control charts can be constructed to alert the analyst to problems, but there is no provision for rejection of results for samples of this type.

8.7.2 Inter-laboratory Quality Control Procedures

An inter-laboratory quality control program serves to select and evaluate methods, characterize their precision and accuracy, and provide data for evaluating both laboratory and analyst performance. Specific objectives of this program are to:

o Measure the precision of reproducibility of methods of analysis within various programs.

o Identify interference in different sampling environments.

o Measure the precision and accuracy of results between laboratories.

o Provide a mechanism for evaluation and/or certification of laboratories and analysts.

o Detect weak, improper, or impractical methodology.

o Detect training needs and upgrade laboratory performance.

o Assist laboratories or programs in obtaining new resources.

The inter-laboratory quality control program is referred to as the Accuracy and Performance Audit* Programs by the Quality Assurance Office Region V. This program was briefly referred to in Section 4.2.1.

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*Audit - A check made by the QAO or its representative to determine the reliability of a specific step in a measurement. For example, a check on the flow of a Hi-Vol air sampler, the sensitivity of a spectrometer detector and the ability to analyse a blind unknown sample are all audits.

*System Evaluation - An on-site inspection and review of the total quality assurance and quality control program. The inspection will be made by the QAO or its representative.

8.7.2.1 Management of the Accuracy and Performance Audit Programs

The number of programs in water, waste, air and special projects are several. The number of measurement parameters which are utilized in these several programs are numerous. QAO presently manages this program by manual means. All data is evaluated manually, which requires a considerable amount of time. As soon as the last Milestone on page 7 of this document is met the QAO will manage an audit program utilizing ADP programs which will provide the needed level of audits that will assure quality data adequate for the requirements of the data user.

The areas of activities which are covered under this audit program are described below:

1. Audits are to be performed according to frequencies and procedures required by Federal Regulations, EPA Guidelines or Region V Policy (e.g., air audits shall conform to the requirements of 40 CFR Part 58, Appendix A and B). The scope of audit must be determined for each measurement parameter (analyte). A performance audit for the measurement of a given analyte in drinking water would be carried our by mailing a reference sample to a laboratory. The reference sample would contain the analyte in a concentration known to QAO, but unknown to the analyst. The analyte would be measured and the value reported back to QAO. A similar type audit would be performed for sewage treatment plants, laboratories analyzing water from lakes and streams, etc. On the other hand, an audit of a Hi-Vol Sampler would require an individual going to a sampling site, measuring flow rates using two or more reference plates and examine the equipment for maintenance and operating conditions, recording temperature, pressure and other information.

2. Audit materials are available from EMSL-Cincinnati, EMSL-RTP, EMSL-LV and commercial sources. Audit materials may be prepared as needed by QAO in conjunction with Central Regional Laboratory and/or contractors. A repository of reference materials will be maintained by QAO for special substances, substances obtained by contract and from other sources when not available from EMSL. A central reference file will be developed which can be accessed by Automatic Data Processing (ADP). This file will give the facility providing the reference material, the concentration or weight per unit and method used to establish reference value. Reference materials will be referenced to the highest standard available, preferably to National Bureau of Standards. Materials which have assay values which have been

verified by Best Available Technology (BAT) are referred to as SRM's and those which have been assayed by NBS are NBS-SRM. Federal Regulations and EPA Guidelines will be followed in determination of the appropriate SRM. Other materials will be measured by BAT and designated as reference samples.

3. Studies will be conducted as specified in QAO's program plan to accomplish certain objectives relative to audits, reference materials, and methods. Determination of the "true value" of an analyte is at time tenuous. Only an estimate of the "true value" can be made for some analytes. The determination or estimation of the value for a reference material will be derived from collaborative testing. These studies will use data from studies such as:

   a. The methodology within the International Joint Commision (IJC) group of analytic systems is not uniform and various methods may be used to measure a given analyte. The results for a reference material analyzed by a variety of methods will be less predictable and the estimate of "true values" less precise. The studies must discern the overall reliability of the methods and identify methods which tend not to measure the analyte. Studies will be used to establish procedures which are uniform for a given measurement principal. The data from such studies require statistical evaluation and at time sophisticated matrix solutions. These statistical evaluations will be processed on ADP.

   b. Methods may be used which are not designated as reference or equivalent methods. Methods for many analytes have not had suitable evaluations and accuracy is not known. Thus, studies will be made by QAO to provide data files on BAT when such information can be obtained. Data files, data systems such as Comnet. EMSL-Cincinnati, etc., will be accessed through a Tektronix 4014 terminal.

   c. Laboratories will be evaluated by performance audits and system evaluations which will include methodology, calibration, training, maintenance and other operations. Audit data and production records will provide a measure of the effectiveness of the quality control program.

4. Independent audits for determination of measurement accuracy will be managed by QAO. The individuals performing audits may be located at the Region V, QAO or in the various S&A district

offices. The audit procedures will be prepared by QAO or if audits are performed by personnel other than QAO those procedures will be approved by QAO.

State and local agencies will develop their audit procedures. Conformance with Federal Rgulations and EPA Guidelines will be determined by QAO. Audit procedures must be reviewed by the appropriate agency on an annual basis and revised as appropriate. Revisions must be approved by QAO.

Each instrument will require an independent audit performed by a State agency, Region V or by contract. The frequency of audits is to be based on requirements of regulations, EPA guidelines or Region V policy.

Some audit results may be reported by ADP terminal as soon as ADP is functional, to QAO for storage in suitable data files. These audit reports would provide date, time, auditor, analyte, method, instrument (reference method, equivalent non-equivalent), agency name, site number, temperature, pressure, all pertinent technical data and values observed. A written report to the agency and the respective Region V media program manager will be made indicating acceptability of performance and/or corrective action required and expected time required to meet compliance.

The operating agency will acknowledge corrective action and reply by indicating that corrective action was taken or would be completed by a given date. Where corrective action can not be made with existing equipment, intended action must be indicated. The QAO will review the operating agency response for acceptability.

A re-audit will be scheduled by QAO and effectiveness of corrective action verified. Verification will be made by an appropriate QAO staff member or appropriate auditor and reported to QAO.

The final audit report is written up and reported formally. The time of reporting will conform to the QAO program plan requirements. The recipiant of the audit report is the operating agency with copies sent to the respective media program manager.

Reports of unacceptable audits sent to the QAO will automatically be flagged for the particular instrument. Data will be invalidated or held in storage as invalid until corrective actions are completed. This system will improve on the data analysis over the present system because audits are not presently correlated with the data until after summaries have been prepared. Thus, it is necessary to go backwards and delete invalid date at a later date. There has not been a systematic method for correlating audits with site data.

Types of instrument audits which will be made are:

a. Ozone calibration audits. These are resource intensive since a calibration system must be maintained with strict quality control measures. The method of measurement must be by the reference method. The audit requires transporting a primary calibration or a transfer calibrator to a monitoring site. Multi-level calibration checks are made which may require as much as 3 to 4 hours operating time. These audits will be performed by auditors located in the district offices and verified by QAO, or performed by QAO.

   Audit results for all systems in the Region are correlated to estimate a "true value" to define accuracy of ozone measurements in Region V.

   Frequency of ozone audits and the acceptable limits are defined in 40 CFR 53, Appendix A. State and local agencies will be audited with a minimum number of audit frequencies described in the Appendix A of the regulations. Greater frequencies are encouraged to the limit of cost effectiveness. Present auditing levels in Region V are greater than the minimum required in proposed regulations. These levels of audits are considered justified since they improve cost effectiveness. Quicker turn around time on audit reports and improved operations might suggest a lesser frequency, but demonstration of the appropriate levels will result from the evaluations established in this program.

b. Hi-Vol Sampler audits are resource intensive requiring travel to monitoring sites, measurement of several flow rates and evaluation of operations and equipment. Personnel who audit the Hi-Vol Samplers are from the District Offices. Audit is verified by the QAO. Frequency of audits are determined by QAO based on regulations, EPA guidelines or Region V policy. Hi-Vol Samplers are audited by Region V, State and local agencies on a frequency which is greater than that required by 40 CFR 53, Appendix A. Acceptable limits applied in Region V are tighter than required by Appendix A.

Audit reports are transmitted to the QAO for verification. A copy of the report will be provided to the Agency audited and to the appropriate media program manager in Region V. The report records temperature, pressure, unusual instrument findings, site number, Agency name and two or more observed flow rates using reference plates. Reference plates are calibrated by equipment which has been referenced to the highest standards available.

c. Calibration equipment: QAO will procure, operate and maintain calibration equipment required for all measurement parameters. Procedures, specifications, operation manuals, maintenance manuals and spare parts lists will be compiled for use with this equipment and made available for Regional, State and local agency use/or information from the Central Reference Files. List of vendors will be documented for ready reference in order to expedite replacement of equipment. Calibrations will be made on various instruments for measurement of critical pollutants, particularly continuous monitors. QAO will maintain quality control records on these instruments.

d. Audits on detector sensitivity for spectrometers, pH meter accuracy, etc., will also be made on an instrument-by-instrument basis.

8.7.2.2 Management of the On-Site System Evaluation of Total In-House, Federal, State and Local Agency, Contractor, Grantee Monitoring Program

The Quality Assurance Office has total responsibility for managing the system evaluation program. A system evaluation is an on-site inspection and review of the quality control program used for the total measurement system for each specific monitoring program conducted by a Federal, State or local agency. For convenience, some items that each quality control plan must contain (discussed elsewhere in this document) and which will be evaluated are repeated below:

1. Organization and Responsibility - Is the quality control program operational?

2. Sample Collection - Are written procedures available for sample collection and are these followed as documented?

3. Sample Analysis - Are written analysis procedures available and are procedures followed as written?

4. Data Validation - Is a list of criteria for data validation available and it it used?

5. Calibration - Are written calibration procedures available and are procedures followed as written?

6. Performance Evaluations - Are control charts for performance evaluations reviewed and corrections made when indicated?

7. Intralaboratory Tests - Are results from intralaboratory testing reviewed and corrections made when indicated?

8. Preventive Maintenance - Is the preventive maintenance schedule being followed as recommended in the QA plan?

The results of the system evaluation is documented by the QAO for presenting a visual picture of the performance of the program to see if the minimum requirements of the Region's Quality Assurance Plan are being met. If not, deviations are identified and recommendations made for corrections. If corrections are not made, recommendations are made to the appropriate program director for action (eg., withholding grant or contract funds, etc.).

AppendiX 17 depicts how the system evaluation program will function.

A system evaluation will be conducted at all laboratories in Region V funded by EPA engaged in the Clean Water or Clean Air Act, the Safe Drinking Water Act, the Toxic Substance Control Act, and other pertinent Acts. All parameters analyzed for, will be evaluated. The minimum QC elements for these laboratories for several major programs are listed in Appendix 18 and 19 and will be evaluated for compliance with these minimum requirements. The minimum QC elements for the above laboratories engaged in the Clean Air Act monitoring activities are listed in EPA publications 600/9-76-005, 600/4-77-027a, and 600/4-77-127b, "Quality Assurance Handbooks for Air Pollution Measurement Systems", and 40 CFR Part 58, Appendix A and B. Laboratory evaluations will be based on compliance with minimum requirements contained in these documents.

The on-site evaluation programs will be administerd as separate operations. These will be evaluations of the:

a. State principal laboratories and offices (water and wastewater).

b. State principal laboratories and offices (air).

c. Local laboratories analyzing public water supply samples.

d. Local air agency offices.

e. Contract laboratories.

This division of work is required because of the administrative separation of the programs.

9. DATA PROCESSING

The term "data processing" is used to include handling, validation, verification, transmission and storage, and reduction, including software QC considerations as described below. Just as samples and specimens can be destroyed, data can be lost, distorted, misinterpreted, incorrectly transcribed, improperly transposed, overlooked, or subject to other distortions, unless suitable QC procedures are used to protect its integrity.

To obtain meaningful environmental data, the representative sample must be delivered unchanged to the analyst who will develop the needed data by performing the prescribed analysis. The completed (i.e., calculated) results need verification calculations to eliminate outliers or extraneous results and the conversion of acceptable results to some final form for permanent recording of the analytical data in meaningful exact terms. These results are then transferred to a data storage facility for future interpretation and use. All quality control plans must document the mechanism to deal with those requirements listed in 9.1. 9.2 and 9.3. Those mechanisms shall be as stringent as those specified below.

9.1 Data Handling Transmission and Storage

Measurements of the concentration of pollutants, either in the ambient environment or in the emissions from stationary sources, are assumed to be representative of the conditions existing at the time of the sample collection. The extent to which this assumption is valid depends on the sources of error and bias inherent in the collection, handling, and analysis of the sample.

Besides the sampling and analytical error and bias, human error may be introduced any time between sample collection and sample reporting. Included among the human errors are such things as failure of the operator/analyst to record pertinent information, mistakes in reading an instrument, mistakes in calculating results, and mistakes in transposing data from one record to another.

Data handling systems involving the use of computers are susceptible to keypunching errors and errors involving careless handling of magnetic tapes and other storage media. Although it cannot be completely avoided, human error can be minimized.

Data reporting techniques and error sources depend on the type of sensor measurement system. Measurement sensors for pollutant concentration may be classified by their sample collection principle into two categories: (1) Integrated, and (2) Continuous. Pollutant measurement systems may be either integrated or continuous, whereas ambient measurement systems are normally always continuous.

In the integrated sample collection principle, a discrete sample is collected in some medium and is normally sent to a laboratory for analysis. The sampler, field operator and the laboratory analyst can make errors in data handling.

In the continuous sample collection principle, an analytical sensor produces a direct and continuous readout of the pollutant concentration parameter. The readout may be a value punched or typed on paper tape or recorded on magnetic tape. In addition, some continuous measurements systems may also use telemetry to transmit data to a data processing center. Both human and machine errors can occur in data handling in this type of system.

a. Data errors in integrated sampling - For ambient monitoring, the sampler or operator records information before and after the sample collection period. For source emission testing, the operator records information during the sample collection period in addition to before and after it. Acceptance limits should be set for data pertaining to flow rates, etc., and the operator/analyst should invalidate or "flag" sampling data when values fall outside these limits. Questionable measurement results may indicate the need for calibration or maintenance.

   The analyst in the laboratory reads measurements from balances, colorimeters, spectrophotometers, and other instruments; and records the data on standard forms or in laboratory notebooks. Each time values are recorded, there is a potential for incorrectly entering results. Typical recording errors are transposition of digits (e.g., 216 might be incorrectly entered as 126) and incorrect decimal point location (e.g., 0.0635 michg be entered as 0.635). These kinds of errors are difficult to detect. The supervisors must continually stress the importance of accuracy in recording results.

Acceptance limits contained in the measurement method write-up and those shown in the method activity matrix should be used by the analyst to invalidate or "flag" analysis data when values fall outside these limits.

b. Data errors in continuous analyses - Continuous monitoring systems may involve either manual or automated data recording. Automated data recording may involve the use of a data logging device to record data on paper tape or magnetic tape at bench or the remote sampling station, or the use of telemetry to transmit data on-line to a computer at a central facility.

Manual reduction of pollutant concentration data from strip charts can be a significant source of data errors. In addition to making those errors associated with recording data values on record forms, the individual who reads the chart can also err in determining the time average value. Usually the reader estimates by inspection the average concentration. When the temporal variability in concentration is large, it is difficult to determine an average concentration. Two people reading the same chart may yield results that vary considerably.

Persons responsible for reducing data from strip charts should be given training. After a person is shown how to read a chart, his/her results should be compared with those of an experienced analyst. Only after he/she has demonstrated the capability to obtain satisfactory results should a analyst be assigned to a data reduction activity.

Periodically the senior analyst or section chief should check strip charts read by each analyst.

Up to 10 percent of all data reported by each analyst is to be checked by the Quality Assurance Coordinator for errors. If an individual is making gross errors, additional training is to be provided.

Because manual chart reading is a tedious operation, a drop in productivity and an increase in errors might be expected after a few hours. Ideally, and individual should be required to spend only a portion of a day at this task.

The use of a data logging device to automate data handling from a continuous sensor is not a strict guarantee against data recording errors. Internal validity checks are necessary to avoid serious data recording errors. There are two sources of error between the sensor and the recording medium: (1) the output signal from the sensor and (2) the errors in recording by the data logger.

The primary concern about the sensor output is to ensure that only the sensor analog signal and not electronic interferences be converted to a digital readout. Internal validity checks should be planned to "flag" spurious data resulting from electronic interferences.

For a system involving the use of telemetry, it is also necessary to include a validity check for data transmission.

c. Errors in computations - To minimize computational errors, operators and analysts should follow closely the formulae, calculation steps, and examples given for each method, using the calculation instructions and forms provided in the method write-up.

The senior analyst should check the computations of each analyst. Up to 10% of all data reported computations are to be checked by the QA Coordinator for errors.

d. Control charts - Procedures for reviewing data at the operational as well as the managerial levels are to be implemented by data generators (lab and field). Review of measurement results from control samples used during analysis, for example, can indicate out-of-control conditions that would yield invalid data from subsequent analyses, if the conditions are not corrected immediately. At the managerial level, periodic review of data can indicate trends or problems that need to be addressed to maintain the desired level of precision and accuracy. One common tool for statistical analysis of data at both the operational and the managerial levels is the control chart. The major steps in constructing the control chart were outlined in Section 8.7.1.

The control chart provides a tool for identifying the systematic variation (assignable cause) from the system indeterminate variation (random). This technique displays data in a form that graphically compares the variability of all test results with the average or expected variability of small groups of data.

The steps to consider in the application of control charts are the following:

1. Select critical characteristics in the measurement system to audit.

2. If audit (reference) standards are used, obtain the necessary materials.

3. Select the data quality objective to audit:

    a. Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions.

    b. Accuracy - The difference between an average value and the true value when the latter is known or assumed.

4. Choose the audit size and frequency:

    a. Size - i.e., for air analysis, the analysts will often be dealing with samples of 2, which will form most subgroups.

    b. Frequency of subgroup sampling - Changes are detected more rapidly as the sampling frequency is increased. Audit rates of 7-10 percent are recommended for many characteristics shown in the method activity matrices.

5. Set control limits, Control limits (CL) are to be set at 2 times the standard deviation for P

6. The control charts are to be maintained either by the operator/analyst or the supervisor. The control chart should be kept up to date. The QA coordinator is to review the charts on some established frequency. After establishing 15 to 20 data points, the control limits should be reestablished on the basis of these data. If the new control limits are narrower than those recommended, the former is to be used. After this initial calculation, control limits should be recalculated every 3 to 6 months, or whenever significant data trends or shifts become obvious.

    The control chart is actually a graphical presentation of quality control effectiveness. If the procedure is "in control", the results will almost always fall within the established control limits. Further, the chart will disclose trends and cycles from assignable causes that can be corrected promptly.

e. Report Forms - The analytical information reported should include the measured parameters; the details of the analyses such as burette readings, absorbance, wavelength, normalities of reagents, correction factors, blanks; and the reported data values. To reduce errors in manipulation of numbers a general rule is to reduce handling and transposition of date to an absolute mininum. Ideally, a report form includes preliminary information about the sample and its analysis, and the same form is used for the final reporting form for entering of data into a computer. However, if such a set-up is not available the protocol below is to be used to record finalized data.

1. Loose Sheets - Reporting of data onto loose or ring-binder forms is a means of recording data that allows easy addition of new sheets, removal of older data, or collection of specific data segments. However, the easy facility for addition or removal also permits loss or misplacement of sheets, mixups in date sequence, and ultimately questionable status of the data for formal display or presentation as courtroom evidence. Loose sheets are not encouraged.

2. Bound Books - The use of bound books is an improvement in data recording that tends to result in a chronological sequence of data insertion. Modification beyond a simple lined book improves its effectiveness with little additional effort. Numbering of pages encourages use of data in sequence and also aids in referencing data through a table of contents ordered according to time, type of analysis, kind of sample, and identity of analyst.

   Validation can be easily accomplished by requiring the analyst to date and sign each analysis on the day completed. This validation can be strengthened further by providing space for the laboratory supervisor to witness the date and the completion of the analyses.

   A further development of the bound notebook is the commerically available version designed for research-type work. These notebooks are preprinted with book and page numbers, and spaces for title of project, project number, analyst signature, witness signature, and dates. Each report sheet has a detachable duplicate sheet that allows up-to-date review by management without disruption of the notebook in the laboratory.

Bound notebooks can and should be used in routine analytical laboratories. The need for repeated information on sampling and analyses can be answered by use of preprinted pages in the bound notebook.

3. Preprinted Report Forms - Most field laboratories and installations repetitively analyzing fixed parameters develop their own system of compiling laboratory data that may include bound notebooks, but a means of forwarding data is also required. Usually, laboratories design forms to fit a related group of analyses or to report a single type of analysis for a series of samples. As much information as possible is preprinted to simplify use of the form. With loose-sheet, multicopy forms (using carbon or NCR paper) information can be forwarded on the desired schedule while also allowing retention of data in the laboratory. Still, the most common means for recording data in rough form are internal bench sheets or bound books. The bench sheet or book never leaves the laboratory but serves as the source of information for transfer of data to appropriate report forms.

   In most instances the supervisor and anlayst wish to look at the data from a sampling point or station in relation to other sampling points or stations on or in a particular AQCR, river or lake. This review of data by the supervisor prior to release is a very important part of the QC program of the laboratory; however, such reviews are not easily accomplished with bench sheets. For review purposes, a summary sheet can be prepared that displays a related group of analyses from a number of stations. The form should contain space for all of the information necessary for reporting data, the completed form can also be used to complete the data forms forwarded to the computer storage and retrieval system.

   The forms used to report to storage systems provide spaces for identification of the sampling point, the parameter code, the type of analysis used, the reporting terminology. Failure to provide the correct information can result in rejection of the data, or insertion of the data into incorrect parameter fields. As sample analyses are completed, the data values are usually reported in floating decimal form along with the code numbers for identifying the parameter data fields and the sampling point data fields.

4. Plastic-Coated Labels and Forms - A recent addition to good sample handling and data management is the availability of plastic-coated (blank or preprinted) labels, report forms, and bound report books. These materials are waterproof, do not disintegrate when wet or handled, can be written on while wet, and retain pencil or waterproof ink markings though handled when wet.

5. Digital Readout - Instrumental analyses, including automated, wet-chemistry instruments such as the Technicon Auto Analyzer, the atomic absorption spectrophotometer, the pH meter, and the selective electrode meter, provide digital readout of concentrations, which can be recorded directly onto report sheets without further calculation. Programmed calculators can be used to construct best-fit curves, to perform regression analyses, and to perform a series of calculations leading to final reported values.

6. Keypunch Cards and Paper Tape - Because much of the analytical data generated in laboratories is first recorded on bench sheets, then transferred to data report forms, keypunched, and manipulated on small terminal computers (or manipulated and stored in a larger data storage system), there is a danger of transfer error that increases with each data copy. The analyst can reduce this error by recording data directly from bench sheets onto punch cards that can be retained or forwarded immediately to the data storage system. Small hand-operated keypunch machines are available.

7. Automated Laboratory Systems - The use of digital readout, keypunch cards, and paper tape have been overshadowed by the development of customized, fully automated online computer systems that make measurements, calculate results, perform qulaity control, and report analytical data simultaneously from a full range of laboratory instruments. Such systems can contain the following functions:

    a. Manual or automatic sampling and testing of a series of samples, standards, replicates and check samples.

    b. Detection of the measurement signals from the series of samples.

    c. Conversion of signals to concentrations, generation of a standard curve, and calculation of sample values in final units.

    d. Calculation of the deviation and recovery values of the results and indication of acceptance or nonacceptance based on limits established by the analyst.

e. Provision of the output in a form designated by the analyst: dial, paper recording chart, digital readout, cathode ray tube, or printed report form.

The degree of hands-on operation required in the system is specified by the analyst.

If an automated system is properly designed and operated, most calculation and transposition errors are avoided and the proper level of quality control is automatically exerted.

9.2 Data Validation and Verification

Data validation is the process whereby data are filtered and accepted or rejected based on a set of criteria. This involves a critical review of a body of data in order to isolate and locate spurious values. It may involve only a cursory scan to detect extreme values or a detailed evaluation requiring the use of a computer. In either situation, when a spurious value is located, it is not immediately rejected. Each questionable value must be checked for validity. Records of values that are either judged invalid or are otherwise suspicious should be maintained. These records are, among other things, a useful source of information for judging data quality. Validation methods can be manual or by computerized techniques.

a. Manual - Both the analyst and the laboratory supervisor should inspect integrated environmental quality monitoring data. At regular intervals, daily or weekly, results should be scanned for questionable values. This type of validation is most sensitive to extreme values, i.e., either unusually high or low concentrations. These are sometimes called outliers.

The criteria for determining an extreme value are derived from prior data obtained at the particular sampling site (or a similar site if no previous data are available for a site). The data used to determine extremes may be the minimum and maximum concentrations for all prior data from a site. The decision criteria might also be based on minimum and maximum for each season, each month, or each day.

An audit level of 7-10 percent should be established for checking data, i.e., checking 7-10 out of every 100 values.

b. Computerized Techniques - A computer can be used not only to store and retrieve data but also to validate data. Any system for checking extreme values in manaul techniques also apply here. The criteria for extreme values can be refined to be specific for individual hours during the day. For example, with this procedure, an hourly average concentration for carbon monoxide of 15 ppm may not be considered an extreme value for 8:00 a.m. but could be tagged as questionable if it appeared at 2:00 am.

Another indication of possible spurious data is a large difference in concentrations reported for two successive time intervals. The difference in concentrations, which might be considered excessive, may vary from one time to another for the same pollutant. Ideally this difference in concentration is determined through a statistical analysis of historical data. For example, it may be determined that a difference of 0.05 ppm in the SO_2 concentration for successive hourly averages occurs rarely (less than 5 percent of the time). But at the same station the hourly average CO concentration may change by as much as 10 ppm. The criteria for what constitutes an excessive change may also be linked to time of day and pollutant relationships, e.g., high concentrations of SO_2 and O_3 can not co-exist and these data should be considered suspect.

c. Criteria for Determining Acceptability of Data - Reading strip charts is a tedious job subject to varying degrees of error. A procedure for maintaining a desirable quality for data manually reduced from strip charts is important. One procedure for checking the validity of the data reduced by a analyst is to have another analyst or the supervisor check the data. Because the values have been taken from the strip chart by visual inspection, some difference in the values derived by two individuals can be expected. When the difference exceeds a specified amount and the initial areading has been determined to be incorrect, an error should be noted. If the number of errors exceeds a predetermined number, all data for the strip chart are rejected and the chart is read again by a technician other than the one who initially read the chart. The question of how many values to check can be answered by applying one of two techniques.

   1. Application of Acceptance Sampling Techniques - Acceptance sampling can be applied to data validation to determine the number of data items (individual values on a strip chart) that need to be checked to determine with a given probability that all the data items are acceptable. The supervisor wants to know, without checking every data value, if a defined error level has been exceeded. From each strip

chart with N data values, the supervisor can randomly inspect n data values. If the number of erroneous values is less than or equal to c, the rejection criteria, the values for the strip chart are accepted. If the number of errors is greater than c, the values for the strip chart are rejected, and another analyst is asked to read the chart.

2. Sequential Analysis Test Procedure - The typical approach used in performing a statistical test of hypothesis requires the collection of a sample of a fixed size. A statistic is then computed from the sample data and compared with some critical values for that statistic. A decision is then made to accept the hypothesis (H_0) or to accept some alternative hypothesis (H_1). With such a procedure it is necessary to collect the specified sample of observations regardless of the results that may be obtained from the first few observations.

   Sequential analysis requires that a decision be made after each observation or group of observations. This procedure has the advantage that, on the average, a decision can be reached with fewer observations than a fixed sample size requires.

d. Data Validations Procedures and Criteria for the Agency's National Aerometric Data Bank (NADB)

The NADB is a computer storage and retrieval system for aerometric data collected by Federal, State and local air agencies.

40 CFR Part 58.35 specifies the NAMS data submittal requirements to NADB which are lsited below:

a. The requirements of this section apply only to those stations designated as NAMS by the network description required by §58.30.

b. The State shall report quarterly to the Administration (through the appropriate Regional Office) all ambient air quality data and information specified by AEROS Users Manual (EPA-450/2-76029, OAQPS No. 1.2-039) to be coded into the SAROAD Air Quality Data forms. Such air quality data and information must be submitted on either paper forms, punched cards, or magnetic tape in the format of the SAROAD Air Quality Data forms.

c. The quarterly reporting periods are January 1-March 31, April 1-June 30, July 1-September 30, and October 1-December 31. The quarterly report must:

   1. Be submitted within 90 days of the end of each reporting period, and

   2. Contain all data and information gathered during the reporting period.

d. The first quarterly report will be due on or before June 30, 1981, for data collected during the first quarter of 1981.

e. Air quality data submitted in the quarterly report must have been edited and validated so that such data are ready to be entered into the SAROAD data files. Procedures for editing and validating data are described in AEROA Users Manual (EPA-450/276-029, OAQPS No. 1.2-039).

f. This section does not permit a State to exempt those SLAMS which are also designated as NAMS from all of any of the reporting requirements applicable to SLAMS in §58.26.

Highlights from the above documents for data validation are described below:

1. Screening Criteria - In order to draw correct conclusions from the data, validity checks are built into the data handling system. The data must meet predetermined standards with respect to representativeness, instrument averaging time, duration of sampling, and comparability before they are incorporated into NADB. A discussion of each criterion follows:

   a. Representativeness - Data from each monitoring site should characterize ambient levels in an area or neighborhood. For example, a daily average of carbon monoxide calculated from values collected only during the morning rush hour would hardly reflect the true daily averages. The data must be relatively complete over the time interval of interest (for example, day, season, or year) so that such biases can be avoided.

b. Instrument Averaging Time - The data must represent a sample interval of 1 hour or more. Thus, no more than 24 values per day per pollutant are stored. Data for intervals of less than 1 hour are converted to hourly averages before storage.

c. Duration - The data must be collected over a time period of no less than 3 consecutive months so that at least quarterly summary statistics can be calculated.

d. Comparability - Aeromatic data must be maintained in consistent units to permit data submitted by various agencies to be combined into nationwide summaries and evaluation reports. The data must have been acquired by application of standard methodologies.

These four criteria are pertinent to developing meaningful information from the data collected from any monitoring network.

2. Criteria for Completeness. The raw data entering the NADB are checked for completeness (representativenesss). With continuous measurement, the criterion for completeness is that at least 75 percent of the total possible number of observations be present. Figure 9.2 presents the number of observations required by the NADB before summary results are calculated.

   The data within the NADB resulting from intermittent sampling are summarized only if there are at least five samples per quarter. An additional stipulation is that if a month contains no samples each of the other 2 months in the quarter must contain at least 2 samples. Any other distribution of samples over the quarter is acceptable. This is a minimum criterion based on a random biweekly sampling schedule. A more stringent criterion should be applied when the sampling schedule is every third or sixth day.

3. Criteria for Accuracy and Precision - Accuracy and precision data reported with aerometric data are not used to validate data before entry into NADB but are used to interpret the data.

4. Criteria for Handling Data Values below Minimum Detectable Limits - Concentrations below the limit of detection of the instruments employed result in the problem of determining how to report such values so that summary statistics can

Continuous measurement criteria for completeness

Time interval	Minimum number of observations
3-hour running average	3 consecutive hourly observations
8-hour running average	6 hourly observations
24-hour	18 hourly observations
Monthly	21 daily averages
Quarterly	3 consecutive monthly averages
Yearly	9 monthly averages with at least two monthly averages per quarter

Figure 9.2 Criteria for completeness for continuous ambient air monitors for NADB

be calculated. The choice of data values is complicated by the fact that zero, the most likely value to be supplied, cannot be used, especially if geometric parameters are to be calculated.

This problem is handled by inserting a constant, approximately equal to one-half the minimum detectable limit, for each method and analysis technique. This value was chosen after examining the lower end of the cumulative distributions for the various pollutants. Seldom did the log-normal distribution (the distribution most often applied to air pollution data) accurately describe this portion of the data. This may be due in part to the existence of a background level for each pollutant. Use of the midpoint between zero and the detectable limit as the substitute value for concentration levels below the detection threshold seems reasonable. In order to permit consistency from year to year, one minimum detectable value is used for each pollutant even if the minimum detectable limit is changed, unless there is a change by an order of magnitude. Figure 9.2.1 provides an example of the current minimum detectable limits as used by the NADB for each pollutant and the value to be inserted for each value below the minimum detectable limit. These minimum detectable limits are reviewed periodically and changed as required. Each laboratory should determine its own set of minimum detectable limits, based on its own analytical techniques and instruments, to generate pollutant information.

One additional point should be mentioned concerning the use of substituted values for values below the threshold of the method. When more than 25 percent of the measured levels are below the minimum detectable quantity, no statistics are computed from the data. This contraint guards against the possibility of biasing the computed statistics. Furthermore, at least 50 percent of the measurements in a set of data must be above the minimum detectable concentration before a frequency distribution of the values can be prepared.

5. Criteria for Handling Data with Negative Values - For the purpose of generating true pollutant values, negative pollutant concentrations imply that there is not enough of the pollutant present for the instrument to detect

Pollutant	Method of Analysis	Minimum detectable limit, ug/m^3	Substitution factor ug/m^3
Suspended particulate	Gravimetric	1.0	0.5
Fluoride	Specif ion electrode	0.05	0.025
Nitrate	Reduction-dizao	0.05	0.025
Sulfate	Colorimetric	0.5	0.025
Carbon monoxide	Nondispersive infrared	573	286
Sulfur dioxide	Pararosaniline, bubbler	5	2.5
Sulfur dioxide	Pararosaniline, continuous	26	13
Ozone	Chemiluminescence	20	10
Total oxidant	Colorimetric	20	10
Nitrogen dioxide	Arsenite, bubbler	5	2.5
Nitrogen dioxide	TGS-ANSA, bubbler	5	2.5
Nitrogen dioxide	Chemiluminescence, continuous	9	4.5
Nitrogen dioxide	Colorimetric, continuous	9	4.5

Figure 9.2.1 Minimum detectable limits, by pollutant and method of analysis, used in NADB in 1975

above the noise limit specified for the instrument. When negative values occur, they should be regarded as being below the detection limit for the method and treated in the same manner, i.e., assigned a value one-half the minimum detectable limit.

9.3 Data Reduction (Including Software QC Considerations)

The effectiveness of the quality assurance program will be determined by monitoring improvements in the reliability of reported QC data. On a regular basis, the QAO will critically evaluate all reported QC data for each reporting unit. It is believed that this approach will provide a data base to more accurately evaluate and improve measurement performance. The QAO will use a number of statistical techniques and ADP to measure performance, which are briefly described below. These techniques have been reviewed extensively elsewhere in this document.

a. Summary Statistics - Summary statistics such as the mean and the standard deviation will be used to simplify the presentation of data and at the same time to summarize essential characteristics.

b. Frequency distributions - Frequency distributions such as normal and log-normal distributions will be used to present relatively large data sets, such as the daily concentrations of suspended particulates in ambient air over a long period of time, i.e., six months.

c. Estimation and testing procedures - Statistical estimation and testing procedures will be used to make inferences concerning the conceptual population of measurements made under the same conditions based on a small sample of data. An example would be the estimation of the average pH of a large number (population) of filters based on a sample (lot) of pH readings for seven filters.

d. Outliers - Outliers, i.e., unusally large or small values, are identified by appropriate statistical tests for outliers. These statistical tests are useful, for example, in identifying gross errors in data handling procedures.

e. Audit data - Statistical methods for treating performance audit data and for presenting the results in terms of bias and precision will be used.

f. Replication, repeatability, and reproducibility tests - The identification of sources of measurement error within and among laboratories is one of the important functions of the Quality Assurance Office. Statistical methods will be used to identify these measurement errors.

Quality control audit data from up to 50 laboratories using several hundred different methods to monitor several hundred different contaminants must be monitored on a continuing basis. This workload requires extensive use of computer technology and considerable skill in interpreting the results. The statistical software package that has been identified in the description of needs, once developed and implemented will be able to handle this large volume of data and generate reports that describe data quality in non-technical language for the data users. Reports will also be produced for laboratory and field personnel describing their performance relative to that of other groups using similar measurement procedures. It is intended that these reports will generally be in a graphical format and include a listing of all supporting results.

The QAO data files will be protected in the ADP system per the file protection protocol for the ADP system. Files will not be accessible to other offices. Programs will be stored as "Declared Files". The original documentation of the software programs will be placed in the permanent files of the QAO in case there is ever a need to refer back to this documentation. Updated print-outs will also be maintained on all file data in case the data is lost due to some hardware malfunction of the ADP system.

10. CORRECTIVE ACTIONS

Corrective actions are of two types:

a. On the spot or immediate - This is the process of correcting malfunctioning equipment.

In a quality assurance program, one of the most effective means of preventing trouble is to respond immediately to reports from the operator of suspicious data or equipment malfunctions. Application of proper corrective actions at this point can reduce or prevent the collection of poor quality data. Established procedures for corrective actions are available in the methods if the performance limits are found to be exceeded (either through direct observation of the parameter or through review of control charts). Specific control procedures, calibration, pre-sampling or pre-analysis operational checks, etc., are designed to detect instances in which corrective action is necessary. A check-list for logical alternatives for tracing the source of a sampling or analytical error is provided to the operator. Trouble shooting guides for operators (field technicians or lab analysts) are generally found in instrument manufacturer's manuals. On-the-spot corrective actions routinely made by field technicians or lab analysts should be documented as normal operating procedures, and no specific documentation other than notations in operations logbooks need to be made. However, logbooks are to be made available to QAO for review during any audit or on-site system evaluation.

b. Long-term Corrective Action - The purpose of long-term corrective action is to identify and eliminate causes of nonconformance or noncompliance with Agency QA requirements, Hopefully, they will be eliminated permanently. To improve data quality to an acceptable level and to maintain data quality at an acceptable level, it is necessary that the quality assurance system be sensitive and timely in detecting out-of-control or unsatisfactory conditions. It is equally important that, once the conditions of unacceptable data quality are indicated, a systematic and timely mechanism is established to assure that the condition is reported to those who can correct it and that a positive loop mechanism is established to assure that appropriate corrective action has been taken. A system of reporting deficiencies and verifying corrective actions identified during the audit and on-site sytem evaluation process has been identified earlier in this document and will not be repeated here.

1. Closed-loop Corrective Action System for Major Problems - Experience in Region V has been that most problems will not disappear until positive action has been taken by management. The significant characteristic of any good management system is the step that closes the loop--the determination to make a change if the system demands it (this is mandated by the Agency's QC requirements and QA regulations).

The following discussion outlines the considerations and procedures necessary to understand and implement an effective closed-loop corrective action system for major problems. Effective corrective action occurs when many individuals and media programs cooperate in a well planned program. There are several essential steps that must be taken to plan and implement a corrective action program that achieves significant results.

Corrective actions should be a continual part of the laboratory system for quality, and they should be formally documented. Corrective action is not complete until it is demonstrated that the action has effectively and permanently corrected the problem. Diligent follow-up is probably the most important requirement of a successful corrective action system.

Figure 10.1 illustrates the sequence of activities involved in operating a closed-loop corrective action system.

10.1 QA Management

Sections 1 and 8.7.2 describes the management responsibility for Region V's Quality Assurance Program. Feedback channels are identified for keeping informed of the performance of all monitoring systems in Region V funded by EPA. Procedures are also identified to monitor the performance of the monitoring systems. Elements of the program have been developed from the QAO Functional Statement, Agency Regulations and requirements which serve as the foundation of Region V's policy statement, have been approved by the Regional Administration, making this program binding on the Region. Goals have been identified (including resources) to accomplish the objectives of this program.

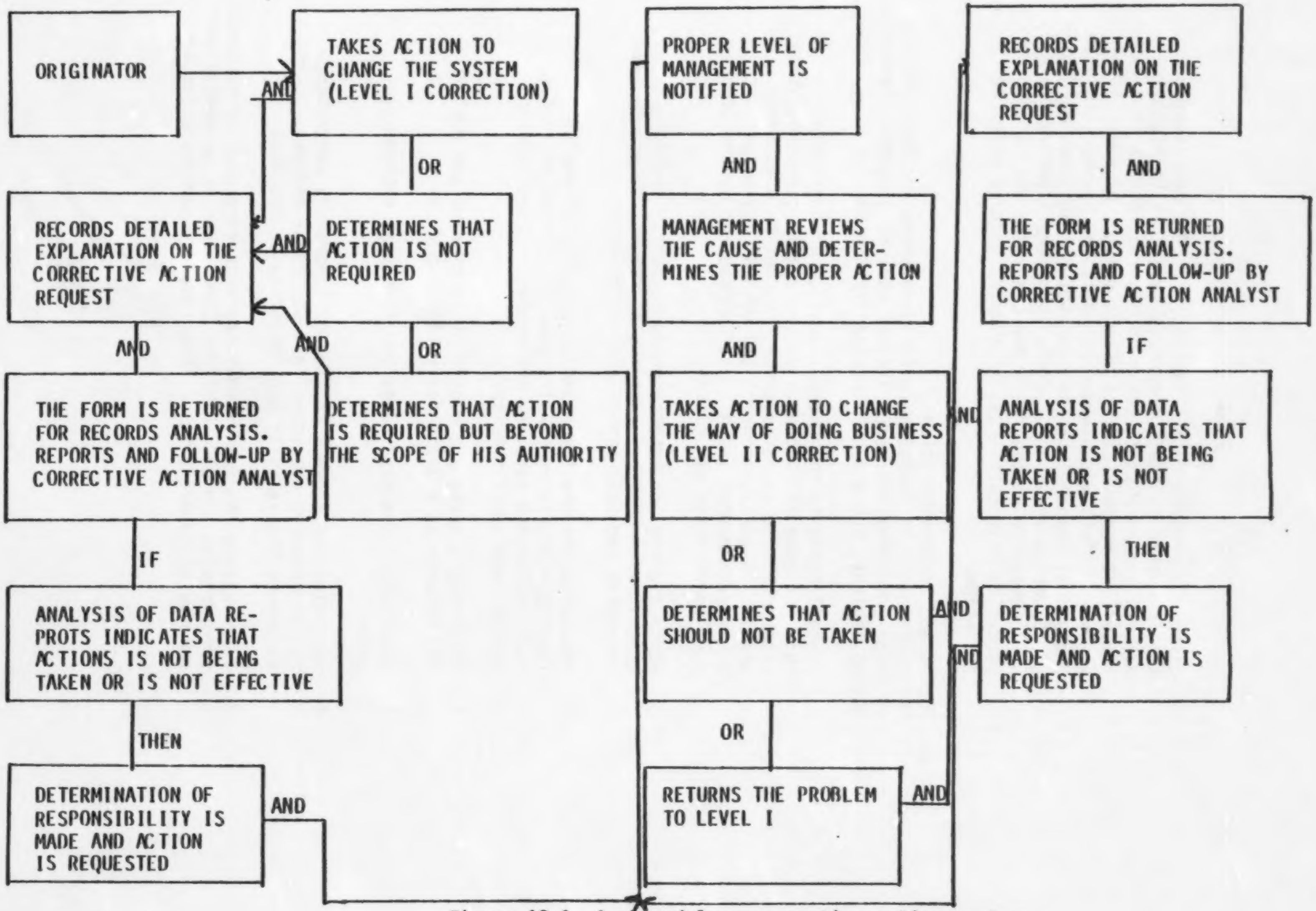
10.2 QC Management

Each monitoring activity shall document and implement a quality assurance policy approved by management to assure that sufficient quality control activities are maintained to assure data credibility for each monitoring project. Each monitoring project shall designate a Quality Assurance/control coordinator (preferably full-time) to be responsible for the environmental QC program, coordinators can be appointed for specific monitoring activities, i.e., Air coordinator, water coordinator.

a. Qualifications

1. The coordinator should have as a minimum a bachelor's degree in physical science, chemistry, biology or microbiology, with at least five years of experience in his respective discipline. In addition, the coordinator must have actively worked in a environmental quality laboratory for at least two years. Experience in statistical quality control techniques and/or academic courses in mathematics and statistics is also highly desirable.

2. The coordinator maintains close liaison with the appropriate EPA Regional Analytical Quality Assurance Coordinator, and is responsible for the overall quality


ORIGINATOR
AND
TAKES ACTION TO CHANGE THE SYSTEM (LEVEL I CORRECTION)
OR
DETERMINES THAT ACTION IS NOT REQUIRED
AND
OR
DETERMINES THAT ACTION IS REQUIRED BUT BEYOND THE SCOPE OF HIS AUTHORITY
AND
RECORDS DETAILED EXPLANATION ON THE CORRECTIVE ACTION REQUEST
AND
THE FORM IS RETURNED FOR RECORDS ANALYSIS. REPORTS AND FOLLOW-UP BY CORRECTIVE ACTION ANALYST
IF
ANALYSIS OF DATA REPROTS INDICATES THAT ACTIONS IS NOT BEING TAKEN OR IS NOT EFFECTIVE
THEN
DETERMINATION OF RESPONSIBILITY IS MADE AND ACTION IS REQUESTED
AND
PROPER LEVEL OF MANAGEMENT IS NOTIFIED
AND
MANAGEMENT REVIEWS THE CAUSE AND DETERMINES THE PROPER ACTION
AND
TAKES ACTION TO CHANGE THE WAY OF DOING BUSINESS (LEVEL II CORRECTION)
OR
DETERMINES THAT ACTION SHOULD NOT BE TAKEN
OR
RETURNS THE PROBLEM TO LEVEL I
AND
AND
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AND
RECORDS DETAILED EXPLANATION ON THE CORRECTIVE ACTION REQUEST
AND
THE FORM IS RETURNED FOR RECORDS ANALYSIS. REPORTS AND FOLLOW-UP BY CORRECTIVE ACTION ANALYST
IF
ANALYSIS OF DATA REPORTS INDICATES THAT ACTION IS NOT BEING TAKEN OR IS NOT EFFECTIVE
THEN
DETERMINATION OF RESPONSIBILITY IS MADE AND ACTION IS REQUESTED

Figure 10.1 A closed-loop corrective action system

assurance program in his laboratory. The coordinator should report to the appropriate level: It is highly desirable that this function not be subordinate to an individual responsible for direct conduct of sampling or analyses. This arrangement is workable, however, if the individual responsible for sampling and analyses maintains an objective viewpoint. While the overall program workload will determine whether this position is a full-time or part-time responsibility, it should, in most cases be full-time.

b. Duties and Responsibilities
The coordinator is responsible for developing and implementing an inter-and-intralaboratory quality control program. Specific duties include, but are not necessarily limited to:

1. Participating in the overall quality control plan. This includes all elements of the sampling and analytical programs. The coordinator carries out this activity within EPA quality control and methodology guidelines. Other recommended and accepted procedures can be used to supplement these guidelines.

2. Administering the interlaboratory quality control program as a continuing in-house activity to ensure the integrity and validity of analytical data.

3. Measuring the precision and/or accuracy of analytical results. Providing on-line quality control of samples, i.e., reference samples, duplicates, control charts, spiked, and audit samples.

4. Providing a permanent record of instrument and analyst performance as a basis for evaluating data.

5. Identifying training needs and technical methodology gaps.

6. Upgrading the overall quality of laboratory performance by recommending procedural and personnel changes, as required, to ensure the validity and integrity of the data.

7. Coordinating the inter-and-intralaboratory quality control program with the QAO, Region V, and other governmental and commercial laboratories. This involves participating in

round-robin methodology studies, providing quality control check samples, and performance of check samples to requesting laboratories.

8. Evaluating and discussing the results of activities outlined in Paragraphs 1 through 7 with the appropriate individuals involved. When an analysis is out of control or a discrepancy is noted, the coordinator should be notified and appropriate corrective action should be taken.

c. Competence of Personnel
The coordinator should develop a training program to ensure a minimal level of proficiency. He must recognize variations in ability and provide training to ensure that professional skills are appropriate to the task. Training programs should be administered in order to develop that level of competence which is necessary to carry out assigned functions. Moreover, these programs should be carried out in full cooperation with EPA Region V, Quality Assurance Office.

d. Basic facility and Equipment Requirements
The coordinator should establish basic requirements (equipment, proper facilities, etc.) for operating an environmental laboratory. These requirements should not be included as part of the quality assurance budget. Laboratory facilities should provide an environment free from atmospheric contaminant levels which can affect the desired analyses. The laboratory should be clean, air conditioned and/or heated, and have a well lighted work area. Safety features and other facilities consistent with opeational requirements should be provided.

e. Initial On-Site Laboratory Evaluation
The coordinator will implement his planned quality assurance program with an initial on-site laboratory evaluation. Subsequent performance of analysis on audit samples and participation in split sample program with the EPA regional office should also be required.

11. DATA QUALITY ASSESSMENT

The assessment of data quality is the end result in a comprehensive QC/QA program. Data quality assessment has four basic components: 1) accuracy, 2) precision, 3) completeness, and 4) representativeness.

Each of these items is quantifiable and when suitably combined can produce a numerical coefficient which is numerically proportional to data quality.

A complete assessment of data quality, in terms of the four components, is not possible at this time. However, this is the primary goal of this QA effort. Air data is more advanced that water and wastewater data at this time for such a comprehensive assessment of data quality. However, with the implementation of this plan during FY 80, a numerical assessment will be factored into the FY 81 QA program plan activity. The primary key in this activity is to get all quality control programs developed, approved and implemented.

The four basic components of data quality assessment have been elaborated on in great detail and their requirements are listed elsewhere in this document, but will be summarized below.

11.1 ACCURACY ASSESSMENT

The QA Plan shall require that the accuracy of environmental data be determined and reported provided that certified reference materials are available or that measurements can be traceable to a national standard.

11.2 PRECISION ASSESSMENT

The Region V QA Plan requires that the precision of environmental data be determined on a routine basis and reported to the suitable management authority as spelled out in the QA and QC Management Section of this document (11.1 - 11.2).

11.3 COMPLETENESS ASSESSMENT

The Region V QA Plan requires that the completeness of environmental data be assessed on a routine basis and reported to the suitable management authority based on approved methodology. Where the method is unapproved an alternate test procedure approved by the Regional Administrator, shall be used. In certain specific cases where methodology does not exist, the QAO will request EMSL to specify a methodology for the Agency's use.

11.4 REPRESENTATIVENESS ASSESSMENT

The Region V QA Plan requires that the representativeness of environmental data be assessed on a routine basis and reported to the suitable management authority using approved methodology. Where the method is unapproved, the same protocol specified in 11.3 is to be followed.

11.5 OVERALL DATA QUALITY ASSESSMENT

Overall data quality assessments are to be included with each data report for water and wastewater at the start of FY 81. The overall data quality assessment for air data is presently being reported.

12. DATA QUALITY REPORTS (QC AND QA)

The following types of QC, QA reports are to be prepared by each monitoring group. These reports serve as a indicator of the monitoring group's progress in implementing its Quality Assurance Program, which monitor the various subunits' performance of quality control procedures and achievement of quality assurance goals.

1. Analytical reports. To maintain the required flow of QA and QC information within a monitoring group, individual analysts, operators and laboratories need to prepare QC reports on their monitoring and measurement activities. These reports are forwarded to the QA coordinator, who then writes a QA report for the entire laboratory organization.

2. Field Location reports. QC data for remote monitoring sites must be developed and transmitted, either individually or grouped by location (i.e., sectional or regional), to the QA coordinator.

3. Instrument inspection, calibration and maintenance reports. How the instruments used in monitoring or measurement procedures are inspected and maintained should be explained in a report to the QA coordinator.

4. Reference materials and standards reports. The reference materials used and standards followed must be stated. These reports should cover not only, for example, the purity of chemical reagents, but biological materials (such as a discussion of the availability of a particular plant needed in experiments) as well.

5. Training reports (personnel). Who was given quality assurance and/or quality control training? Did this training take place in-house? How much did this training cost? The training reports will answer these questions.

6. Certification reports. These reports will be generated only for the public water supply laboratory certification program. The procedures for performance evaluation and certification of both laboratories and personnel will be detailed in these reports.

7. Quality assurance reports. Reports detailing the unit's quality control and quality assurance activities should be published on a quarterly basis.

Distribution of and follow-up to these reports for corrective action will be the same as that described for all other types of reports described elsewhere in this document.

13. CHAIN OF CUSTODY

The following procedures have been used successfully, and are presented as suggested procedures insofar as they fulfill the legal requirements of the appropriate State legal authority.

a. Procedures

Quality assurance should be stressed during all compliance monitoring and when reviewing self-monitoring programs, no matter what the reason for the spot check or inspection. Successful implementation of a compliance monitroing program depends heavily on the capability to produce valid data, and on clearly demonstrating such validity. No other environmental monitoring area requires more rigorous adherence to validated methodology and quality control measures.

It is imperative that laboratories and field operations involved in collecting primary evidence prepare written procedures. These procedures should be used whenever evidence samples are collected, transferred, stored, analyzed, or destroyed. A primary objective of these procedures is to create an accurate written record which can be used to trace possession of the sample from the time it is collected through its introduction into evidence.

b. Preparing Samples

The evidence-gathering portion of a survey is characterized by an absolute minimum number of samples required to give a fair representation of the effluent or water body sampled. The quantity and location of samples are determined before the survey.

Prepare chain-of-custody record tags before actual field survey work. Ensure tags contain all possible information to minimize clerical work by field personnel. Also write the source of each sample on the container before starting any field survey work.

Field logsheets used to document field procedures and chain-of-custody, and to identify samples, should be pre-filled in to the extent practicable to reduce repetitive clerical field entries. The sampler or project leader should maintain custody during sampling, using the logbook. Any information from previous studies should be copied (or removed) and filed before the logbook is returned to the field.

Follow explicit chain-of-custody procedures to maintain the documentation necessary to trace sample possession from the time the sample is taken until the evidence is introduced into court. A sample is in your custody if:

- It is in your physical possession; or
- It is in your view, after being in your physical possession; or
- It was in your physical possession and you locked it in a tamper-proof container or storage area.

All survey participants should receive a copy of the study plan and should be familiar with its contents before the survey begins. A pre-survey briefing should be held to inform all participants of the survey objectives, sample locations and chain-of-custody procedures. After all chain-of-custody samples are collected, a debriefing should be held in the field to verify that chain-of-custody procedures have been followed, and to determine if additional evidence samples are required.

c. Collecting Samples

1. Ensure that the smallest possible number of people handle the sample.
2. Obtain stream and effluent samples using standard field sampling techniques. When using sampling equipment, assume it is in the custody of the source being sampled.

3. Attach chain-of-custody record tag to the sample container when the complete sample is collected. Ensure the container has the following information: sample number, time taken, date taken, source of sample (include type of sample and name of firm), preservative, analyses required, name of person taking sample, and witnesses. The front side of the card (which has been prefilled) is signed, timed, and dated by the person doing the sampling. Tags must be legible and filled out in ballpoint (water-proof ink). Secure individual sample containers or group of sample containers using a tamper-proof seal.

4. Take blank samples. Include one sample container without preservative, and containers with preservatives. The laboratory will analyze these contents to verify that no containers are contaminated.

5. Maintain an up-to-date Field Data Record Logbook. Record field measurements and other pertinent information necessary to refresh the sampler's memory if, later on, he takes the stand to testify regarding his actions during the evidence-gathering activity. Maintain a separate set of field notebooks for each survey; store them in a safe place where they can be protected and accounted for at all times. Standard formats have been established to minimize field entries; these include the date, time, survey, type of sample taken, volume of each sample, type of analysis, sample number, preservatives, sample location and field measurements (temperature, conductivity, DO, pH, flow), and any other pertinent information or observations.

   The field sampler signs the entries. The survey coordinator is usually responsible for preparing and conserving the field logbook during the survey. Once the survey is complete, field logs will be retained by the survey coordinator or his designated representative, as a part of the permanent record.

6. The field sampler is responsible for the care and custody of the collected samples until they are properly dispatched to the receiving laboratory, or turned over to an assigned custodian. The field sampler should verify that each container is in his physical possession or in his sight at all times, or is locked so that no one can tamper with it.

7. Colored slides or photographs are often taken to show the outfall sample location and any visible water pollution. Written documentation on the back of the photo should include the photographer's signature, and the time, date, and site location. These photographs can be used as evidence, and are handled by chain-of-custody procedures to prevent alteration.

d. Transfer of Custody and Shipment

1. When transfering the possession of samples, the transferree signs, dates, and times the reverse side of the chain-of-custody record tag or record. Custody tranfers, if made to a sample custodian in the field, are made for individual samples. The chain-of-custody tag or card must be dated and signed by the second person who takes custody. If a third person takes custody, he must follow the same procedure. An additional chain-of-custody tag or card is completed by persons who thereafter, take custody. It is apparent, from this chain, that the number of custodians should be minimal. Additional tags or cards should be numbered consecutively.

2. If a custodian has not been assigned, the field custodian or field sampler is usually responsible for properly packaging and dispatching samples to the proper laboratory for analysis. In that case, the "Dispatch of Sample" portion of the chain-of-custody record tag or card should be properly filled out, dated, and signed.

3. Ensure that samples are properly packed in shipping containers (for example, ice chests) to avoid breakage. Ensure that shipping containers are padlocked for shipment to the receiving laboratory.

4. Include a "Sample Transmittal Sheet" with all packages. The original, and one copy generally accompany the shipment. Mail copies directly to the laboratory, to data management personnel, and to any other responsible agent. The survey coordinator usually retains one copy.

5. If the package is sent by mail, ensure that it is registered with return receipt requested. If package is hand-delivered, record delivery in the logbook. Send receipts from post offices and bills of lading to the laboratory custodians for retention as part of the permanent chain-of-custody documentation.

6. When samples are delivered to the laboratory, and appropriate personnel are not there to receive them, samples should be locked in a secure, tamper-proof area. The same person must unlock the samples and deliver custody to the appropriate custodian.

LABORATORY CUSTODY PROCEDURES

The following procedures are to be used by Region V monitoring activities to provide the chain of possession and custody of any sample offered for evidence, and for which analytical test results amy be introduced into evidence in any environmental case.

The primary objective of these procedures is to create an accurate written record which can be used to trace the possession and handling of the sample from the moment of collection through analysis and its introduction as evidence.

1. The laboratory director will designate one full-time employee (usually the laboratory supervisor) as a sample custodian, and one other person as an alternate. In addition, the laboratory must provide a sample storage area that is secure and can be locked.

2. All samples will be handled by a minimum possible number of persons.

3. Only the custodian will receive incoming samples. If he is absent, the alternate will indicate receipt by signing the sample transmittal sheets and, (as appropriate), the sample tags which accompany the samples. The alternate will retain the transmittal sheets as permanent records.

4. The custodian shall ensure that heat-sensitive, light-sensitive samples, radioactive, or other sampe materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained prior to analysis.

5. Distribution of samples to the section chiefs who are responsible for the laboratory performing the analysis shall be made only by the custodian.

6. The laboratory area shall be maintained as a secured area, restricted to authorized personnel only.

7. Laboratory personnel are responsible for the care and custody of the sample once it is received by them and shall be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses were completed.

8. Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned, tagged sample, should be retained in the custody room until permission to destroy the sample is received by the custodian.

9. Samples shall be destroyed only upon the order of the Laboratory Director, in consultation with previously designated Enforcement officials, or when it is certain that the information is no longer required or the samples have deteriorated. The same procedure is true for tags and laboratory records.

e. EVIDENTIARY CONSIDERATIONS

Reducing chain of custody procedures as well as the various promulgated laboratory analytical procedures to writing will facilitate the admission of evidence under rule 803(6) of the Federal Rules of Evidence (PL. 93-575). Under this statute, written records of regularly conducted business activities may be introduced into evidence as an exception to the "Hearsay Rule" without the testimony of the person(s) who made the record. Although preferable, it is not always possible to have the individuals who collected, kept, and analyzed samples testify in court. In addition, if the opposing party does not intend to contest the integrity of the sample or testing evidence, admission under the Rule 803(6) can save a great deal of trial time. For these reasons, it is important that the procedures followed in the collection and analyses of evidentiary samples be standardized and described in an instruction manual which, if need be, can be offered as evidence of the "regularly conducted business activity" followed by the lab or office in generating any given record.

In criminal cases however, records and reports of matters observed by police officers and law enforcement personnel are not included under the business record exceptions to the "Hearsay Rule" previously cited (see Rule 803(8), P.L. 93-595). It is arguable that those portions of the compliance inspection report dealing with matters other than sampling and analysis results come within this exception. For this reason, in criminal actions records and reports of matter observed by field investigators may not be admissible and the evidence may still have to be presented in the form of oral testimony by the person(s) who made the record or report, even though the materials come within the definition of business records. In a criminal proceeding, the opposing counsel may be able to obtain copies of reports prepared by witnesses, even if the witness does not refer to the records while testifying, and if obtained, the records may be used for cross-examination purposes.

Admission of records is not automatic under either of these sections. The business records section authorizes admission "unless the source of information or the method or circumstances or preparation indicate lack of trustworthiness," and the caveat under the public records exception reads "unless the source of information or other circumstances indicate lack of trustworthiness".

Thus, whether or not the inspector anticipates that his or her compliance inspection report will be introduced as evidence, he or she should make certain that the report is as accurate and objective as possible.

14. SPECIFIC GUIDANCE

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APPENDIX 1

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

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Page 1 of 23

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-235 Air Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: SLAMS Network Monitoring
OBJECTIVE: Assure that QA Programs are a part of, and adequate for all EPA grants and contracts which include monitoring equipment.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Review six State grant requests and requests for equipment. Recommend approval/disapproval. Monitoring equipment must meet requirements specified in the regulations.	Young/Kocal	5 days after receipt of request	QAO's role not addressed in the approved work plan package. Coordinate with TSB Air.			
2. Review local Agency grant request and request for equipment. Recommend approval/disapproval. Monitoring equipment must meet requirements specified in the regulations.	Young/Kocal	5 days after receipt of request	Coordinate with TSB Air.			

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APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-235 Air Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: Implement Air Monitoring Quality Assurance
OBJECTIVE: To insure that the quality of data collected, reported or used by the Agency is properly documented, sufficiently accurate and precise to meet Agency needs.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Develop and implement a interim Regional quality assurance program for air that has EDP capability for a more efficient approach to data handling and provide a more accurate database to evaluate and improve analytical performance of all Region V, Federal, State and local laboratories generating air quality monitoring data. Implement final revision of National Quality Assurance Plan when received.	Young/Adams	Target date of 1/15/80 no later than 3/15/80	This action step will be performed in conjunction with DU B-224, DU B-303, DU B-209, DU c-215 and DU B-241. Development and implementation of EDP part of program depends on funding of the software package (programs) requested in the QAO FY 80 budget. Implementation of National QA programs depends on receipt from Headquarters.			
2. Evaluate QC policy and programs for all six Region V States per criteria specified in 40 CFR Part 58, including management and documentation of the program.	Young/Moran	20 days after receipt of request	Coordinate with TSB/Air.			
3. Develop system to consolidate TSB/Air and QAO reviews of State air Agency monitoring and QA programs.	Adams	12/30/7	Crosscut issue with TSB/Air.			

Form PEB 101

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

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ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-235 Air Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: Implement Air Monitoring Quality Assurance OBJECTIVE:				TRACKING/MONITORING		
KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	DATE COMP.	CHK BY	CORRECTIVE ACTION
4. Review for approval or disapproval six Region V 105 grants for Agency quality assurance requirements.	Young/Moran	5 days after receipt of grant	Linkage - TSB/Air and AMM.			
5. Manage eleven EMSL-RTP Inter-Lab Surveys for Region V State and local laboratories. Data is reviewed and proficiency of lab is measured. Follow-up letters are provided for problems identified to the State and the local Agency laboratories.	Young/Moran	10 days	Coordinate with EMSL-RTP.			

Form PEB 101

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-235 Air Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: Data Reporting and Analysis
OBJECTIVE: To ensure that all data reported out of the S&A Division and Region V States meets Agency requirements as spelled out in the regulations for sufficient quantity and suitable quality.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Perform four data quality assessment audits of the data reporting and analysis function of the TSB, Air. This function will be performed on a quarterly basis with report to management.	Young/Moran	10 days after end of each quarter	QAO's role not addressed in the approved work plan package. However, part of QAO's responsibility is evaluating S&A monitoring activities of which data reporting and analysis is part of any monitoring project. Resources not provided for this activity. However, it will be piggy backed on to other DU A-235 activity that is funded.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-235 Air Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: Implement Air Monitoring Regulations
OBJECTIVE: To standardize all air monitoring in Region V States by implementation of 40 CFR Part 58 Appendix A. Comparable data requires reference to a common standard. QAO's system will assure standard & comparable data.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Coordinate and overview fifteen audits of monitors operated by State and local Agencies which are to be provided by EMSL-RTP. Reports and follow-up will be provided by QAO to the States and local Agencies. (EMSL-RTP has funds to provide this technical assistance to Region V. Plan has been finalized by QAO).	Young/Kocal	10 days after receipt of RTP finding	This step is based on EMSL-RTP following through on this commitment to Region V. QAO is seeking to have RTP schedule this activity the 3rd quarter of FY 80. The entire effort will be coordinated with EMSL-RTP.			
2. QAO will conduct a maximum of twelve additional audits (as 1 above) with follow-up.	Young/Kocal	Six in 3rd Qtr Six in 4th Qtr	Report turn around time will be 20 days after the date of audit. Completion of this action step depends on full funding of QAO's travel request budget for FY 80 air activity.			

APPENDIX 1

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

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ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-235 Air Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: Implement Air Monitoring Regulations on Quality Assurance
OBJECTIVE: Implement a system audit program designed to upgrade local agency monitoring activities compatability with EPA/State program requirements

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Evaluate and approve local agency monitoring programs for laboratory capability, quality assurance practices and data validation. Approximately 30 local Agency's are located in Region V.	Young/Horan	15 days after State Eval. 21 days after local agency eval.	The QAO will evaluate the State's effort in evaluating local agency's in their respective State as part of the State program evaluation (A-325 Implement Air Monitoring QA). If deficiencies are found, an on-site system evaluation of the local agency will be conducted. QAO estimate that deficiencies will be found in 80% (24) of the local agencies as a result of the State review which will entail 24 on-site evaluations. Completion of this action step is based on QAO receiving the level of travel funds requested for FY 80 air activity.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-305 Air Enforcement
MANAGER: James H. Adams, Jr.

ACTIVITY: Case Development Inspections
OBJECTIVE: A major function of the QAO is to conduct a vigorous audit program to insure data reliability for all enforcement (legal) proceedings.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
Seven ambient air quality studies have been identified for FY 80. A maximum of three audits (on-site evaluations) will be conducted during the life of each study for conformance with QA requirements and for data validity.	Young/Kocal	30 days after onsite or 20 days after receipt of DO's audit report for verification	This activity will be coordinated with the DO's, TSB Air and the Enforcement Division. A maximum of 21 audits/evaluations will be conducted. On-site evaluations (audits are dependent on QAO receiving the level of travel funds requested for FY 80 air activity.			

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APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-305 Air Enforcement
MANAGER: James H. Adams, Jr.

ACTIVITY: Unleaded Gas Inspections
OBJECTIVE: Ensure data quality is adequate for enforcement (legal) proceedings.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Review sample data and QC data for validity. Provide follow-up for problem correction where problems are identified in the audit process. A maximum of six audits will be conducted.	Young	10 days after receipt of QC data	The QAO will coordinate this activity with the DO's and CRL. Sample and QC data will be evaluated two times (2nd and 3rd quarters) for each DO and the CRL.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): A-305 Air Enforcement
MANAGER: James H. Adams, Jr.

ACTIVITY: PSD Monitoring
OBJECTIVE: To insure monitoring activity conform to the requirements specified in Appendix B, 40 CFR, Part 58.

				TRACKING/MONITORING		
KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	DATE COMP.	CHK BY	CORRECTIVE ACTION
1. PSD monitoring has not been addressed. Air Enforcement has informed the QAO that there will be approximately 40 PSD's. The authority for approving the PSD monitoring can be delegated to the States. Illinois, Indiana and Wisconsin have made some progress in this program, no authority has been delegated to any Region V States. Thus, Region V has this responsibility. PSD permits requires the implementation of Appendix B, 40 CFR Part 58. The evaluation of each PSD is time consuming. Additionally, there is a requirement for audit.	Young/Kocal	30 days after receipt of documts or 30 days after onsite	Coordinate with TSB/Air and Air Enforcement. Completion of this action step is based on receiving the level of travel funds requested for air activity.			

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APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-209 Dredge and Fill
MANAGER: James H. Adams, Jr.

ACTIVITY: Manage the QA Activity for the Ecological Evaluation of Proposed Discharge of Dredged or Fill Material into Navigable Waters
OBJECTIVE: Provide a strong QA program to assure contract laboratory(s) are providing quality data for use in decision making that accurately describe the characteristics/concentration of constituents in the samples submitted to the laboratory.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. The QAO will manage an interagency agreement with the Corps of Engineers in which one IPA will be funded by the Corps for QAO to perform the complete QA function for a maximum of eight laboratories under contract to the Corps or a lesser QC function for a larger number of labs. Included in this function are the on-site evaluations.	IPA/Adams	21 days after onsite	This activity is coordinated with the North Central Division of the Corps of Engineers. The first priority is to fill the IPA slot.			
2. Manage reference sample program. EDP evaluation of data with follow-up for problems.	IPA/Adams	15 days after study results are avail.	This activity will be coordinated with EMSL-Cincinnati.			

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APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-224 Ambient Water Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: State and Internal Quality Assurance Used for Management Decisions
OBJECTIVE: Provide a strong QA program to assure that water quality monitoring data will meet Agency needs and requirements.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Develop and implement a interim Regional quality assurance program for water and wastewater that has EDP capability for a more efficient approach to data handling and provide a more accurate database to evaluate and improve analytical performance of all Region V Federal, State and local laboratories generating water quality monitoring data. Implement final revision of national quality assurance plan when received.	Payne/Adams	Target date of 1/15/80 No later than 3/15/80	This action step will be performed in conjunction with DU A-235, DU B-303, DU B-209, DU C-215, and DU B-241. Development and implementation of EDP part of program depends on funding of the software package (program) requested in the QAO FY 80 budget.			

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APPENDIX I (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-224 Ambient Water Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: State and Internal Quality Assurance used for Management Decisions
OBJECTIVE:

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
2. Review for approval or disapproval six Region V State 106 grants for agency QA requirements.	Payne/OPFT	10 days after receipt of applic.	Coordinate with TSB/Water.			
3. Evaluate six State laboratories and the CRL for conformance with Agency requirements (Annual evaluations). Follow-up will be provided in areas where problems are identified. Some labs will require quarterly follow-up evaluations for performance improvement. Reports will be prepared.	Payne/Sturino/ Marion	30 days of onsite	Coordinate with TSB/Water. Completion of this action step is based on QA receiving the level of travel funds requested for FY 80 ambient water quality monitoring activity.			
4. Evaluate six State 106 quality assurance programs for approval or disapproval. Follow-up provided in areas where problems are identified. The CRL's on-going QA program is also evaluated..	Payne/Marion	30 days of receipt of program	Coordinate with TSB/Water. This step will be performed in conjunction with Step 3 of this activity.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-224 Ambient Water Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY: OBJECTIVE: State and Internal Quality Assurance Used for Management Decisions

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	CHK BY	CORRECTIVE ACTION
5. Manage the P.E. and reference sample program for Region V 106 laboratories. Data evaluation will be conducted. Follow-up on problem areas identified will be provided (5 studies involving 15 State labs, CRL, a undetermined number of NPDES dischargers and several commercial labs engaged in analyzing NPDES samples).	Payne/Long	15 days after receipt of data eval. from EMSL	Will coordinate with EMSL-Cincinnati.			
6. Evaluate, approve or disapprove the quality assurance requirements in all ambient water contracts, grants and interagency agreements. Approximately 23 - 208 agencies are involved, plus any other 106 contracts, grants and interagency agreements. On-site evaluation of the laboratory(s) are conducted to insure that a quality assurance plan is operational and all equipment, supplies and personnel necessary for successful completion of the project are available.	Payne/Adams	21 days after receipt of all documents and onsite where required	Will coordinate with respective project officer. Cross cut issue with EEB Aban. Sites and A&HM Waste Mgt. Activities. Resources for this DU (B-224) totals 2 MY(s) (1 PFT, 1 OPFT). This resource will not be enough to complete all of the action steps to meet the objective of this activity. Additional resources will be needed to complete this action step per the requirements of the Agency. Completion of this action step also depends on total funding of QAO's travel request for this activity.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-224 Ambient Water Quality Monitoring
MANAGER: James H. Adams, Jr.

ACTIVITY:
OBJECTIVE: State and Internal Quality Assurance Used for Management Decisions

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
7. Manage the P.E. and reference sample program for 208 laboratories. Data evaluation will be conducted. Follow-up on problem areas identified will be provided.	Payne/Long/Marion	15 days after receipt of data eval. from EMSL	Coordinate with EMSL-Cincinnati.			
8. Provide technical review and processing of a maximum of ten alternate test procedures for approval or disapproval.	Payne	10 days after receipt of EMSL's recomm.	Coordinate with EMSL-Cincinnati.			

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APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-303 Water Quality Enforcement
MANAGER: James H. Adams, Jr.

ACTIVITY: Surface Cases for Federal Enforcement
OBJECTIVE: A major function of the QAO is to conduct a vigorous audit program to insure data reliability for all enforcement (legal) proceedings.

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. At the request of Enforcement, conduct a maximum of ten on-site evaluations for laboratory(s) engaged in analyzing NPDES samples for conformance with Agency QA requirements and data validity.	Payne/Marion	21 days after onsite	This action step will be coordinated with the Enforcement Division and DO's. Completion of this action step depends on total funding of QAO's travel request.			
2. Provide a quality control program for a maximum of two special studies requested by Enforcement. Evaluate reliability of reported data and defend analytical methods.	Payne	21 days after receipt of required documts	This action step will be coordinated with the Enforcement Division.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): C-215 Public Water Supply Management
MANAGER: James H. Adams, Jr.

ACTIVITY: Provide Technical Capability for Implementation of the National Interim Primary Drinking Water Regulations
OBJECTIVE: To insure that laboratories have the capability to perform analytical measurements of all contaminants specified in the NIPDWR (40 CFR 141 and 142).

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
1. Develop and implement a Regional quality assurance program for public water supply that has EDP capability for a more efficient approach to data handling and provide a more accurate database to evaluate and improve analytical performance of all Region V Federal, State and local laboratories generating public water supply monitoring data. Implement final revision of national quality assurance plan when received.	Payne/Adams	Target date of 1/15/80 no later than 3/15/80	This action step will be performed in conjunction with action DU A-235, DU B-224 and DU B-241. Development and implementation of EDP part of program depends on funding of the software package (program) requested in the QAO's budget.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): C-215 Public Water Supply Management
MANAGER: James H. Adams, Jr.

ACTIVITY: Provide Technical Capability for Implementation of the National Interim Primary Drinking Water Regulations
OBJECTIVE:

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	CHK BY	CORRECTIVE ACTION
2. Review for approval or disapproval of six Region V State public water supply grants for laboratory capability and minimum grant QA requirements.	Payne/Long	Dates will be those specif. by grant PO	Will coordinate with Water Supply Branch.			
3. Manage certification program for State principal laboratories (16 State labs involved).	Payne	21 days	Will coordinate with Water Supply Branch. This is an on-going technical exercise that may or may not generate a report. Technical assistance is the main objective until the Agency's final regulations are implemented, which then will entail on-site evaluations for compliance.			
4. Evaluate six local laboratory certification programs, managed by primacy States for conformance with Agency requirements for local certification programs.	Payne/Marion	21 days after onsite	Will coordinate with Water Supply Branch. This step is linked to Step 6 of this activity. Completion of this step depends on total funding of QAO's travel request for this activity.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): C-215 Public Water Supply Management
MANAGER: James H. Adams, Jr.

ACTIVITY: Provide Technical Capability for Implementation of the National Interim Primary Drinking Water Regulations
OBJECTIVE:

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
5. Finalize pending interim certification activity for six State principal labs.	Payne/Adams	21 days after all defics. have been correct	Will coordinate with Water Supply Branch			
6. Provide a minimum of one on-site evaluation visit to each Region V State principal laboratory. Issue reports.	Payne/Sturino/ Marion	21 days after onsite	Will coordinate with Water Supply Branch. Also done in conjunction with Step 4 above.			
7. Perform data quality assessment evaluation of the CRL. This function will be performed on a quarterly basis (part of QAO's responsibility is evaluating S&A monitoring activities which CRL's analytical activities are part of the Division's monitoring activities).	Payne/Long	16 days after end of each qrtr.	Coordinate with CRL.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): C-215 Public Water Supply Management
MANAGER: James H. Adams, Jr.

ACTIVITY: Provide Technical Capability for Implementation of the National Interim Primary Drinking Water Regulations
OBJECTIVE:

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING		
				DATE COMP.	CHK BY	CORRECTIVE ACTION
8. Manage the P.E. and reference sample program for Region V PWS laboratories. Data evaluation will be conducted. Follow-up on problem areas identified will be provided.	Payne/Long/Marion	10 days after receipt of data analy. from EMSL	Coordinate with EMSL-Cincinnati.			
9. Maintain interim certification program for local chemistry laboratories in the non-primacy State of Indiana. A maximum of five labs will be evaluated for possible interim certification.	Payne/Sturino/Young	21 days after lab has met all minimum requirements.	Coordinate with Water Supply Branch. Completion of this objective depends on total funding of QAO's travel request for this activity.			
10. Maintain interim certification program for ten local chemistry laboratories in the primacy States of Michigan and Wisconsin. A maximum of ten labs will be evaluated for possible interim certification.	Payne/Sturino/Young	21 days after lab has met all minimum requirements.	Coordinate with Water Supply Branch. Completion of this objective depends on total funding of QAO's travel request for this activity.			

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APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): C-215 Public Water Supply Management
MANAGER: James H. Adams, Jr.

ACTIVITY: Provide Technical Capability for Implementation of the National Interim Primary Drinking Water Regulat ons
OBJECTIVE:

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	CHK BY	CORRECTIVE ACTION
11. Provide workshop for standardizing metal analyses for Region V public water supply laboratories.	Payne	3/31/8	Coordinate with CRL			
12. Provide workshop for upgrading organics analyses for Region V public water supply laboratories.	Payne/Sturino/ Young	4/30/8	Coordinate with CRL. Completion of this step depends on the availability of Sturino and CRL's organic facilities.			
13. Process a maximum of five alternate test procedure requests for approval by labs analyzing PWS samples.	Payne -	10 day after receip of EMSL's commen s	Coordinate with EMSL-Cincinnati.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

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ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-241 Great Lakes (QAO's role not addressed in the approved wotk plan package)
MANAGER: James H. Adams, Jr.

ACTIVITY: Quality Assurance for Lake Monitoring
OBJECTIVE: Provide a strong QA program to assure contract and Agency laboratories are providing quality data for use in decision making that accurately describe the characteristics/concentration of constituents in the samples submitted to the lab.

	KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	TRACKING/MONITORING CHK BY	TRACKING/MONITORING CORRECTIVE ACTION
1.	Develop and implement a Regional QA program for Great Lakes monitoring program that has EDP capability for a more efficient approach to data handling and provide a more accurate database to evaluate and improve analytical performance of all laboratories generating Great Lakes monitoring data. Implement Agency final revision of national QA plan when received.	Payne/OPFT	Target date of 1/15/80 no later than 3/15/80	This step will be performed in conjunction with DU A-235, DU B-224 and DU C-215. Development and implementation of EDP part of program depends on funding of the software package requested in the QAO's budget.			
2.	Review, approve or disapprove QA plans, sample collection, preservation holding time, and methodology prior to funding of any GLNPO monitoring projects. A maximum of ten projects shall be reviewed.	Payne/OPFT	21 days after receipt of packet	Coordinate with GLNPO.			

APPENDIX 1 (Continued)

QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-241 Great Lakes
MANAGER: James H. Adams, Jr.

ACTIVITY: OBJECTIVE: Quality Assurance for Lake Monitoring

KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	TRACKING/MONITORING DATE COMP.	CHK BY	CORRECTIVE ACTION
3. Manage PE/QC sample program for a maximum of ten laboratories on a quarterly basis. EDP data review and follow-up provided where problems are identified.	Payne/OPFT/ Long	10 days after data analys.	Coordinate with GLNPO. Linkage with the Data Quality Work Group, IJC.			
4. Conduct on-site evaluations of ship board lab and a maximum of ten contract labs for compliance with contract/grant QC and methodology requirements. Based on previous problems, some contract labs will have to be evaluated on a quarterly basis.	Payne/Young/ OPFT	21 days after onsite	Coordinate with GLNPO. Linkage with the Data Quality Work Group, IJC. Completion of this step depends on total funding of QAO's travel request.			

APPENDIX 1 (Continued)

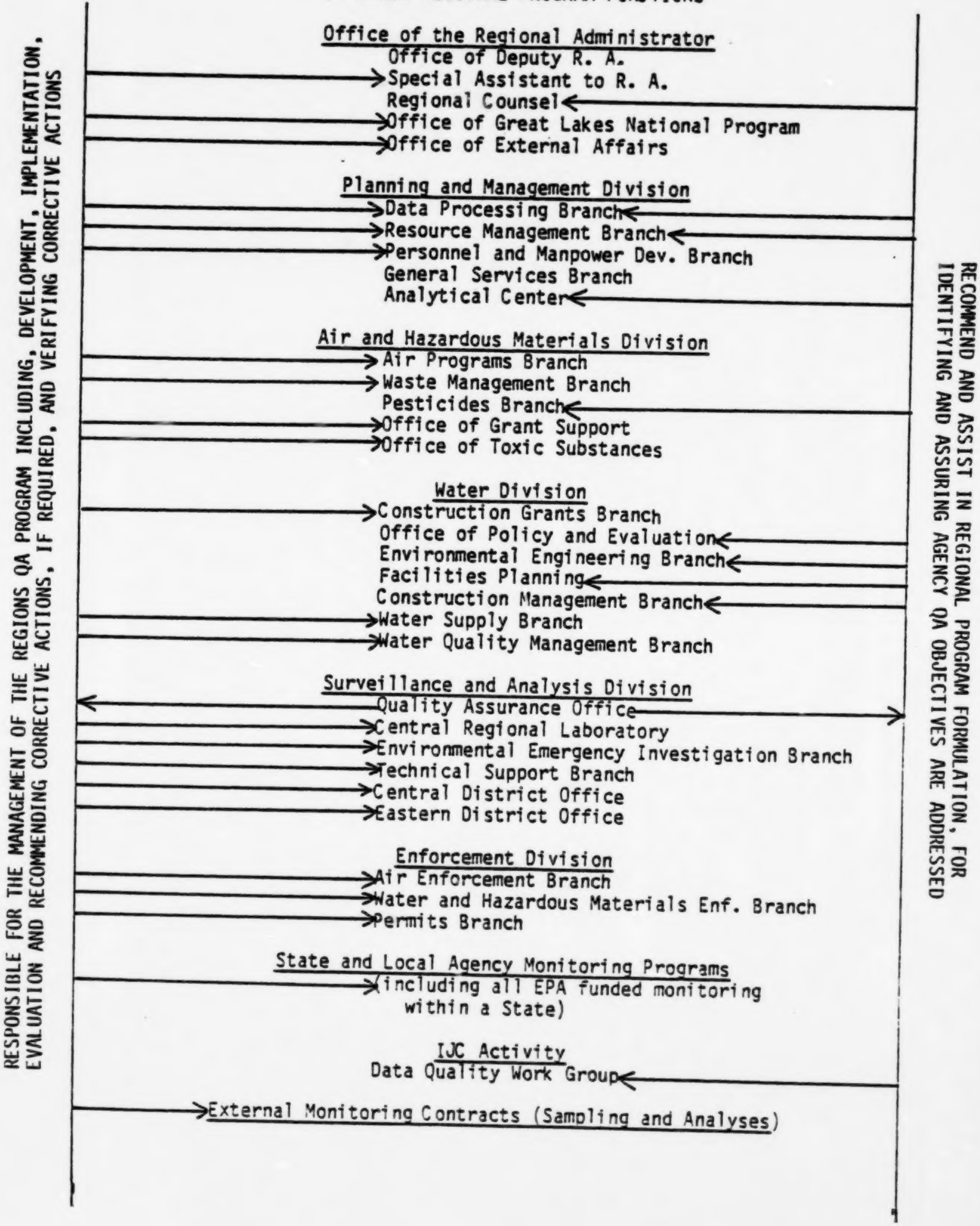
QUALITY ASSURANCE OFFICE
FY 80 WORK PLANS

ORGANIZATION: Quality Assurance Office, S&A Division
DECISION UNIT(s): B-241 Great Lakes
MANAGER: James H. Adams, Jr.

ACTIVITY: Quality Assurance for Lake Monitoring OBJECTIVE:				TRACKING/MONITORING		
KEY ACTION STEPS	ASSN. RESPONSIBILITY	DUE DATES	INTER/INTRA ORGANIZATIONAL PREREQUISITES/LINKAGES/ASSUMPTIONS	DATE COMP.	CHK BY	CORRECTIVE ACTION
5. Particiapate in the activities of the Data Quality Work Group, IJC. These activities relate to work group round robin appraisals, meetings on data quality, Quality Assurance, method standardization and the fish and sediment program.	Adams	N/A	Linkage with CRL and the GLNPO.			

APPENDIX 2

RELATIONSHIP OF THE QUALITY ASSURANCE FUNCTION TO OTHER REGIONAL PROGRAM FUNCTIONS -



RESPONSIBLE FOR THE MANAGEMENT OF THE REGIONS QA PROGRAM INCLUDING, DEVELOPMENT, IMPLEMENTATION, EVALUATION AND RECOMMENDING CORRECTIVE ACTIONS, IF REQUIRED. AND VERIFYING CORRECTIVE ACTIONS

RECOMMEND AND ASSIST IN REGIONAL PROGRAM FORMULATION, FOR IDENTIFYING AND ASSURING AGENCY QA OBJECTIVES ARE ADDRESSED

Office of the Regional Administrator
Office of Deputy R. A.
→ Special Assistant to R. A.
Regional Counsel ←
→ Office of Great Lakes National Program
→ Office of External Affairs

Planning and Management Division
→ Data Processing Branch ←
→ Resource Management Branch ←
→ Personnel and Manpower Dev. Branch
General Services Branch
Analytical Center ←

Air and Hazardous Materials Division
→ Air Programs Branch
→ Waste Management Branch
Pesticides Branch ←
→ Office of Grant Support
→ Office of Toxic Substances

Water Division
→ Construction Grants Branch
Office of Policy and Evaluation ←
Environmental Engineering Branch ←
Facilities Planning ←
Construction Management Branch ←
→ Water Supply Branch
→ Water Quality Management Branch

Surveillance and Analysis Division
← Quality Assurance Office →
→ Central Regional Laboratory
→ Environmental Emergency Investigation Branch
→ Technical Support Branch
→ Central District Office
→ Eastern District Office

Enforcement Division
→ Air Enforcement Branch
→ Water and Hazardous Materials Enf. Branch
→ Permits Branch

State and Local Agency Monitoring Programs
→ (including all EPA funded monitoring within a State)

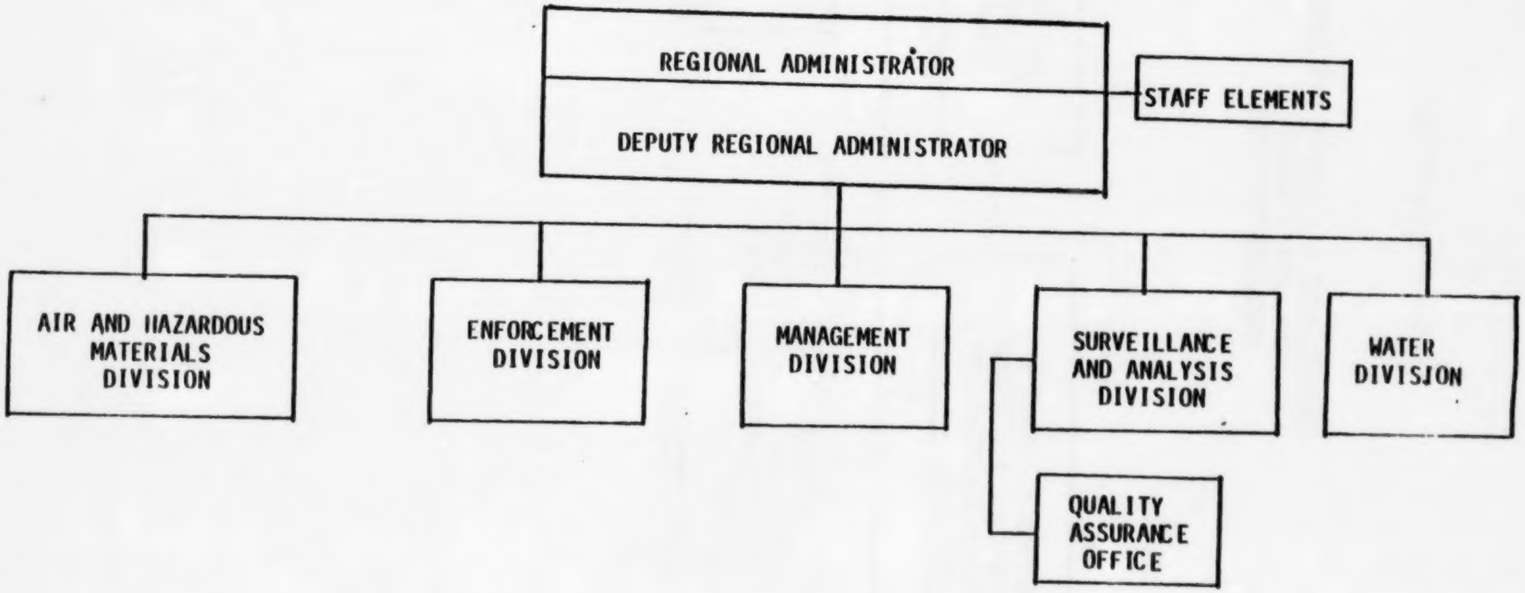
IJC Activity
Data Quality Work Group ←

→ External Monitoring Contracts (Sampling and Analyses)

APPENDIX 3

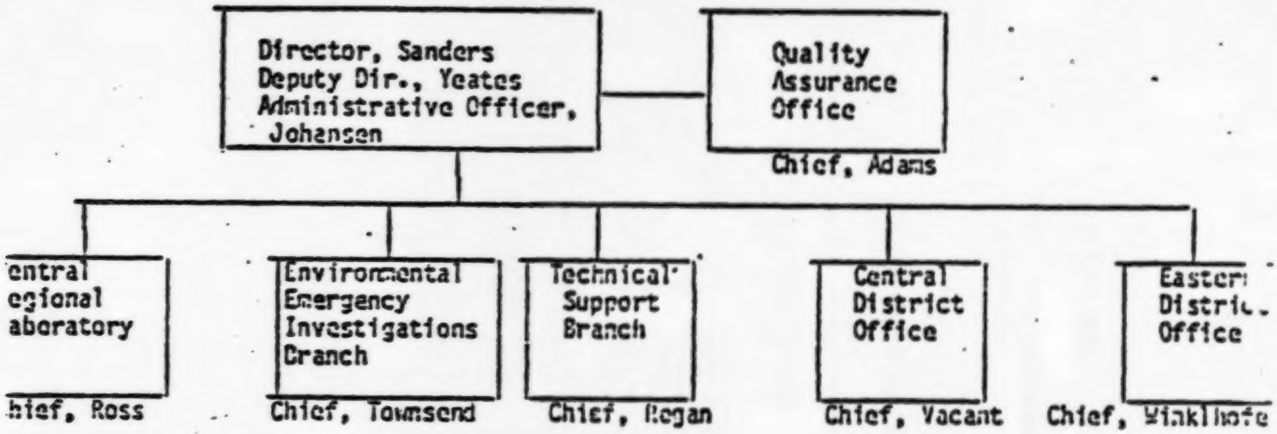
ORGANIZATIONAL STRUCTURE
REGION V

U.S. ENVIRONMENTAL PROTECTION AGENCY


REGIONAL ADMINISTRATOR
DEPUTY REGIONAL ADMINISTRATOR
STAFF ELEMENTS
AIR AND HAZARDOUS MATERIALS DIVISION
ENFORCEMENT DIVISION
MANAGEMENT DIVISION
SURVEILLANCE AND ANALYSIS DIVISION
QUALITY ASSURANCE OFFICE
WATER DIVISION

APPENDIX 3 (Continued)

SURVEILLANCE & ANALYSIS DIVISION
U.S. EPA - REGION V
ORGANIZATION CHART



Director, Sanders
Deputy Dir., Yeates
Administrative Officer, Johansen
Quality Assurance Office
Chief, Adams
entral egional aboratory
hief, Ross
Enviromental Emergency Investigations Branch
Chief, Townsend
Technical Support Branch
Chief, Regan
Central District Office
Chief, Vacant
Easter Distri Office
Chief, Winklhofe

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best available copy.

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APPENDIX 4

STATE OF WISCONSIN
DEPARTMENT OF NATURAL RESOURCES
BUREAU OF AIR MANAGEMENT
AIR MONITORING SECTION

QULITY ASSURANCE MANUAL

PROCUREMENT

Procurement Testing Procedures

The following guidelines are to be used when evaluating continuous monitors prior to purchase. These are not purchase specifications. Articles relating to vendor responsibility and warranty obligations will be included in purchase specification guidelines. This is a description of parameters that must be considered and tested when evaluating monitors prior to purchase.

Pre-purchase instrument evaluation will consist of several parts: 1) a preliminary elimination process based on data gathered by DNR concerning user experience, vendor provided performance test results, and instrument advantages and disadvantages; 2) equipment testing of instruments, selected as a result of screening of data gathered in #1, to assure the instruments perform as stated - this testing will be performed by DNR personnel; and 3) final considerations of equipment usability in DNR's network, vendor cooperation and desirable features, which will further narrow down the number of instruments. Monitors to be considered for evaluation must have been designated by EPA as reference or equivalent methods. No equipment will be approved for purchase without first having completed the evaluation process outlined here. Final selection of instruments to be purchased by DNR will be based on the degree to which the monitor exceeds minimum specifications, the performance test results, purchase and annual operations costs, and availability and cost of service/repair by contract/demand.

NEED ANALYSIS

To begin the analysis, the DNR group undertaking the instrument evaluation must prepare a needs analysis report which analyzes the application for which the instruments will be used and determines which instrument characteristics will best fit the application. For example, will the instrument be used for background monitoring (list the ambient levels expected) or point source monitoring (expected ambient levels are higher). The following parameter needs must be defined in this report:

1. expected concentration range
2. threshold concentration
3. anticipated gas stream composition
4. response time
5. maintenance requirements
6. portability requirements

This report is to be prepared and circulated to each DNR group who will be affected by the instrument purchase, as well as to the Quality Assurance Coordinator, for comments. After comments are received and the report revised, the specifications and user review can take place.

SPECIFICATION AND USER REVIEW

This phase of the evaluation is a weeding out process. As we have neither the manpower nor space for extensive instrument testing, only a small number of instruments will be chosen for such testing. This initial evaluation is to be the means of choosing which instruments will be tested in-house. If more than one measurement principle is listed as a reference or equivalent method (as is the case with SO continuous methods) at least one instrument from each measurement principle must be considered in this initial evaluation. The following information must be gathered for each instrument that is evaluated, and a report prepared on the results of this data gathering phase.

1. Request instrument operating manuals from each manufacturer and review them. Check and compare measurement principles, performance characteristics and relative complexity of operation. List advantages and disadvantages of each.

2. User experience - The manufacturer will be contacted to supply a list of users. EPA should also be contacted for names of any dissatisfied users. Agencies or industries with prior field experience with each particular instrument will be contacted for their opinion of the instrument's mechanical, electronic and chemical dependability (confidence in instrument data), ease of working with the instrument, user experience with the vendor, vendor responsiveness, cost of operation, and instrument downtime. At least two users must be contacted for each instrument evaluated. Attached is a copy (Figure 1) of the questions to be covered when talking to the users.

3. Performance testing - Manufacturer shall provide written results of their equivalency testing, to be evaluated for precision, accuracy, interferences, etc.

4. Vendor cooperation - Each vendor will be contacted and evaluated as to his/her: a) willingness to comply with the terms of the pre-purchase arrangement (our in-hour testing) and purchase contract specs, b) factory and local representative expertise, support, and facilities, c) instrument warranty terms, d) willingness to supply all information required to operate, maintain and repair the instrument.

5. Required support equipment - Determine what is needed in terms of supporting electronics, gas cylinders, etc., for each instrument evaluated, and the availability and cost of such. Which of these items do we already have, their adequacy and their age. Which items must be purchased and the cost of the purchase should be included.

6. Annual operating costs - Approximate cost of parts, reagents, electronics, gas cylinders, manpower support, and a list of high and low motality parts for each instrument evaluated.

7. Conformity of each instrument to existing DNR instrumentation systems (both the vans and the permanent continuous monitoring stations).

   a. manifold
   b. data acquisition system
   c. rack mounting
   d. calibrators

The information in the report will be organized into tables for purposes of comparison; each parameter listed above is to be scored (based on its relative importance as determined in the needs analysis) and a summary chart of comparative scores will be drawn up. These tables and charts will be circulated for comment and recommendations to key persons within the air monitoring program who are knowledgeable with instrumentation. Any instrument or manufacturer not favorably rated in this phase of evaluation will be excluded from further testing. Recommendations for no more than three instruments to be tested in depth in DNR labs, will be made by representatives of each monitoring group and the quality assurance coordinator.

INSTRUMENT TESTING

As a result of the specification and user review, up to three monitors will be tested in-house by DNR personnel.

The purpose of this testing is to:

1. Obtain a working knowledge of each instrument - how easy it is to use, how well it performs, and what problems we might expect with it.

2. Verify that certain crucial equivalency testing parameters are indeed met. Equivalency test results are provided to EPA by the manufacturer and are not verified by EPA. Some users have found that the equivalent designated instruments that they have purchased are not meeting these performance specifications.

3. There are differences in instrument performance among instrument manufacturers whose instruments pass the equivalency specifications. Some instruments just pass the testing, while others have performance that is vastly superior to the equivalency specifications.

The following instrumental tests are to be conducted on all continuous monitors being considered for purchase. Figure 2 is an example of how the results of the testing should be reported.

I. Range

A. Definition

Nominal minimum and maximum concentrations which a method is capable of measuring.

B. Test Procedure

1. Allow the instrument to warm up as per manufacturer's instructions.

2. Construct a calibration curve showing the test analyzer's response over at least 95 percent of the required range.

3. Allow the instrument to run for 24 hours before performing any further tests.

II. Noise Test

A. Definition

Noise is the short-term deviation in output signal which is not the result of changes in input concentration. It is essentially the standard deviation. Noise is an inherent property of an instrument arising from imperfect electronics, mechanical stresses, quality of optics, etc. Noise levels are critical as they set limits on useful measurement levels, and the lower detectable limit is often defined as twice the noise level.

B. Test Procedure

1. Allow sufficient time for the test analyzer to warm up and stabilize.

2. Connect an integrating-type digital voltmeter (DVM) suitable for the test analyzer's output, and accurate to three significant digits, to measure the analyzer's output signal. Also connect the analyzer to an appropriate strip chart recorder.

3. Measure zero air for 60 minutes. Use the range setting specified in Table I. The recorder should be set for 0 to 1 volt full scale. During this 60-minute interval, record 25 readings at 2-minute intervals.

4. Convert each DVM reading or strip chart recording to concentration units (ppm) by reference to the test analyzer's calibration curve. Label the converted DVM readings $r_1, r_2, \ldots, r_{25}$.

5. Calculate the standard deviation, S, as follows:

$$S(\text{ppm}) = \sqrt{\frac{\Sigma(r_i)^2 - 1/25(\Sigma r_i)^2}{24}}$$

6. Let S at zero ppm be S_0; compare S_0 to the noise specification given in Table I.

7. Repeat steps (3) through (6) using a recorder output of either 0 to 1MV or 0 to 5MV. The baseline on the strip chart should be at 50% of full scale, so that positive and negative deviations can be observed. Compare S_{MV} to the noise specification as given in Table I.

C. Lower Detectable Limit

Definition - The minimum pollutant concentration which produces a signal of twice the noise level.

1. Test Procedure

   a. Allow sufficient time for analyzer to warm up and stabilize. Measure zero air for at least 15 minutes and record the stable reading in ppm as B_Z.

   b. Generate and measure for at least 15 minutes a pollutant test atmosphere concentration equal to the value for the lower detectable limit specified in Table I.

   c. Record the test analyzer's stable indicated reading, in ppm, as B_L.

   d. Determine the lower detectable limit (LDL) as LDL = $B_1 - B_Z$. Compare $B_L - B_Z$ to $2S_0$.

Table I - EPA Performance Specifications for Automated Methods

Performance parameter	Units	Sulfur dioxide	Photo-chemical oxidants	Carbon monoxide	Nitrogen dioxide
1. Range	Parts per million	0-0.5	0-0.5	0-50	0-0.5
2. Noise	do	.005	.005	.50	.005
3. Lower detectable limit	do	.01	.01	1.0	.01
4. Interference equivalent					
Each interferent	do	±.02	±.02	±1.0	±0.02
Total interferent	do	.06	.06	1.5	.04
5. Zero drift, 12 and 24 hour	do	±.02	±.02	±1.0	±.02
6. Span drift, 24 hour					
20 percent of upper range limit	Percent	±20.0	±20.0	±10.0	±20.0
80 percent of upper range limit	do	±5.0	±5.0	±2.5	±5.0
7. Lag time	Minutes	20	20	10	20
8. Rise time	do	15	15	5	15
9. Fall time	do	15	15	5	15
10. Precision					
20 percent of upper range limit	Parts per million	.01	.01	.5	.02
80 percent of upper range limit	do	.015	.01	.5	.03

1. To convert from parts per million to ug/m^3 at 25°C and 760 mm Hg, multiply by M/0.02448, where M is the molecular weight of the gas.

III. Zero Drift, Span Drift, Lag Time, Rise Time, Fall Time and Precision Test

(Also may indicate voltage variation and ambient temperature sensitivity)

A. Definitions

Zero Drift - This value is the change in response to zero pollutant concentration over a 24 hour period of continuous unadjusted operation.

Span Drift - This value is the percentage change in response to pollutant concentrations of 80% of scale and 20% of scale over a 24 hour period of continuous unadjusted change.

Lag Time - The time interval between a change in pollutant concentration input and a corresponding change in scale readings.

Rise Time - The time interval between an increase in pollutant concentration input and 95% response to a new concentration level.

Fall Time - The time interval between a decrease in pollutant concentration input and 95% response to the new concentration.

Precision - Precision is defined as a variation about the mean of repeated measurements of the same pollutant concentration expressed as one standard deviation about the mean.

B. Test Procedure - The monitor should be set up in such a manner that the voltage and temperature may be controlled (or recorded) and, if possible, altered experimentally to levels the monitor may be expected to encounter. If the instrument is to be housed in a tightly controlled environment, the monitor need be tested only in a duplication of that environment. This test procedure need only be performed once if the instrument is to be used in a controlled environment; at least three test runs must be performed at varying environmental conditions if the instrument will be subject to voltage and temperature fluctuations at a monitoring site. In either case, more testing should be done if the instrument responds irregularly. During this procedure no manual adjustments to the electronics, gas or reagent flows, other than those specified by the test, or as part of a required periodic maintenance program, is to be performed.

1. Day 1

The instrument shall be operated at 115 volts and at 25°C.

1. Allow sufficient time for instrument warm-up and stabilization. Adjust the zero baseline to 5 percent of full scale. Recalibrate if necessary. (Usually if the span check indicates a span drift in excess of the value listed in Table I.)

2. Arrange to generate test atmospheres as follows:

Test Atmosphere	Pollutant Concentration (Percent)
	URL = Upper range limits
A_0	Zero gas
A_{20}	20 ± 5 of URL
A_{30}	30 ± 5 of URL
A_{80}	80 ± 5 of URL
A_{90}	90 ± 5 of URL

Set chart speed at 2 inches/hr.

3. Measure A_0 until a stable reading is obtained. Record reading as $Z1_0$. Note the clock time on the strip chart.

4. Measure A_{20} until a stable reading is obtained. Record reading as M^1_0. Note the clock time on the strip chart.

5. Measure A_{80} until a stable reading is obtained. Record reading as S^1_0. Note the clock time on the strip chart.

6. Sample A_0 until reading is less than 15 percent of full scale. A stable reading is not required.

7. Measure A_{20}. Record stable reading as P_1.

8. Sample A_{30}. A stable reading is not required.

9. Measure A_{20}. Record stable reading as P_2.

10. Sample A0. A stable reading is not required.

11. Measure A_{20}. Record stable reading as P_3.

12. Sample A_{30}. A stable reading is not required.

13. Measure A_{20}. Record stable reading as P_4.

14. Sample A_0. A stable reading is not required.

15. Measure A_{20}. Record stable reading as P_5.

16. Sample A_{30}. A stable reading is not required.

17. Measure A_{20}. Record stable reading as P_6. Note the clock time on the strip chart.

18. Measure A_{80}. Record stable reading as P_7.

19. Sample A_{90}. A stable reading is not required.

20. Measure A80. Record stable reading as P_8. Set chart speed at 4 inches/hr.

21. Measure A_0. Record stable reading as L_1.

22. Quickly switch test analyzer to measure A_{80}. Mark recorder chart at exact time of switch.

23. Measure A_{80}. Record stable reading as P_9.

24. Sample A_{90}. A stable reading is not required.

25. Measure A_{80}. Record stable reading as P_{10}.

26. Measure A_0. Record stable reading as L_2.

27. Measure A_{80}. Record stable reading as P_{11}.

28. Sample A_{90}. A stable reading is not required.

29. Measure A_{80}. Record stable reading as P_{12}. Note the clock time on the strip chart.

30. Measure A_0. Record stable reading as Z_1. Note the clock time on the strip chart.

31. Measure A_{20}. Record stable reading as M_1.

32. Measure A_{80}. Record stable reading as S_1.

33. Zero Drift

$$\text{Zero Drift} = Z^1{}_0 - Z_1$$

Report the Elapsed Testing Time as Measured in Steps (3) and (30).

34. Span Drift

(a) at 20% URL

$$\text{Span Drift} = \frac{M_n - M_o^1}{M_o^1} \times 100\%$$

where:

$$M_n = \frac{1}{6} \sum_{i=1}^{6} P_i$$

Report the Elapsed Testing Time as Measured in Steps (4) and (17).

(b) at 80% URL

$$\underline{\text{Span}}\ \underline{\text{Drift}} = \frac{S_n - S_o^1}{S_o^1} \times 100\%$$

where:

$$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$$

Report the Elapsed Testing Time as Measured in Steps (5) and (29).

35. **Lag Time**

Determine from the strip chart, the elapsed time in minutes between the mark made in step 22 and the first observable (2 x noise level) response.

36. **Rise Time**

Calculate 95 percent of reading P_9 and determine from the recorder chart the elapsed time between the first observable (2 x noise level) response and a response equal to 95% of P_9.

37. **Fall Time**

Calculate 95 percent of $(P_{10} - L_2)$ and determine the elapsed time in minutes between the first observable decrease in response following P_{10} and the response equal to 95 percent of $(P_{10} - L_2)$.

38. **Precision**

Calculate precision (P_{20} and P_{80}) as follows:

(a)
$$P_{20} = \sqrt{\frac{1}{5}\left[\sum_{i=1}^{6} P_i^2 - \frac{1}{6}\left(\sum_{i=1}^{6} P_i\right)^2\right]}$$

$$P_{80} = \sqrt{\frac{1}{5}\left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6}\left(\sum_{i=7}^{12} P_i\right)^2\right]}$$

2. **Day 2**

Obtain a stable zero air reading. Record. Introduce a test atmosphere of 80% of scale. Allow instrument to run and record at this level for 24 hours. At the end of the 24 hour period, return to zero air and obtain a stable reading. Report the ppm value for the first hour (X_1) and for the last hour (X_2). Report span drift (80%) as $\frac{X_2 - X_1}{X_1} \times 100$

3. Day 3

After obtaining and recording the zero air reading, introduce a 20% of scale test atmosphere. Allow instrument to run and record at this level for 24 hours. Return to zero air and record the stable reading. Report the ppm value for the first hour (Y_1) and for the last hours (Y_2).

Report span drift (20%) as $\frac{Y_2 - Y_1}{Y_1} \times 100$

4. Day 4

Repeat day 2 procedures, except allow instrument to run for 48 to 72 hours.

5. Day 6

Repeat day 4 procedures, except use zero air.

If the instrument will be operated under conditions of fluctuating temperatures and voltages, repeat this test procedure (beginning with Day 1) at least two more times, altering ambient temperatures and voltage levels to settings the instrument is likely to encounter in the field.

IV. Interference Test

A. Definition - Interference is the positive or negative effect of a substance, other than the pollutant being measured, as reflected in instrument response.

B. Test Procedure - The test procedure will vary depending on the instrument and its potential interferences. The procedures to be used will be written by the QA Coordinator (in conjunction with the testing group) prior to the beginning of the testing phase.

V. Flow Rate Measuring Device - Factors to be taken into consideration are the accuracy of the device, ease of calibration (either in or out of the sampling line), ease of adjustment and flow rate drift. Data from this test should be recorded as in Figure 3.

A. Test Procedure -

1. Calibrate the flow rate controller, as specified in the instrument operation's manual, with a transfer standard of known high accuracy (such as a wet test meter, soap bubble meter or mass flow meter). Thereafter, run a daily flow rate check of one or more points to check the flow rate controller's stability. Report the maximum % deviation in the flow rate calibration.

2. Record the flow rates each day, as indicated on the instrument's flow rate measuring devices for each parameter of

interest (H_2 flows, sample flows, etc.). Report the maximum % variability in the flow rate readings for each parameter measured.

For further details regarding any of the above tests, please refer to the February 18, 1975 Federal Register - Ambient Air Monitoring Reference and Equivalent Methods Part II.

VI. Calibration Drift

When all testing is complete, run a multipoint calibration of the analyzer. DO NOT ADJUST ZERO OR SPAN SETTINGS ON THE INSTRUMENT. Compare with the initial calibration as follows:

A. Determine the slope and intercept for each calibration; X = ppm, Y = instrument reading.

B. Using the slope and intercept for each calibration determine the ppm values at each instrument reading from 10 to 100% in units of 10 (see Figure 5).

C. Determine the percent differences for each ppm value obtained in B. Assume the initial calibration value is the "true value."

D. Report the average percent difference as:

$$\% \text{ Diff} = \frac{\sum_{i=1}^{n} d_i}{n}$$

Note that the absolute values of the percent differences are used.

E. Report the maximum percent deviation observed in the region in which ambient concentrations will fall. Report this data on Figures 4 and 5. Instructions for completing Figure 4 precede the figure.

A report must be prepared which includes all the original data, strip charts, calculations and calibrations. In addition, the calculated data - span drifts, calibration drifts, etc., should be organized into tables for purposes of comparison. The following areas determined in the "Specification and User Review" and in the "Instrument Testing" should be included in this report.

1. Vendor Cooperation

    a. willingness to comply with terms of DNR prepurchase and purchase specifications.

    b. factory and local representative expertise, support and facilities. Factory training of technicians who will be required to service and repair equipment.

    c. possibility of direct factory (original equipment supplier) purchase of parts. Specify parts available only from vendor.

   d. instrument delivery time for all ordered monitors

   e. condition of the monitor when received for "instrument testing"

   f. warranty terms

2. Required support equipment - What is needed in terms of supporting electronics, gas cylinders, etc. and availability of such. Detail which items we already own and which would have to be purchased.

3. Annual operation's cost - Approximate cost of parts, reagents, electronics, gas cylinders and manpower support.

4. Operations Manual

   a. ease of comprehension

   b. completeness (including wiring blue prints)

5. Ease of Access for:

   a. instrument repair

   b. routine maintenance

   c. hook up, either free standing or rack mounted

   d. routine calibration

   e. of knobs, switches, and dials

6. Conformity to Existing Instrumentation Systems

   a. manifold

   b. data acquisition system

   c. rack mounting

7. Aesthetic Appeal

Consideration of where and how instrument is to be used should be kept in mind in making a subjective evaluation. Where problems are specific to a certain use of the instrument (ex. if used in the vans it's a problem) specify this in describing the problem.

The report should be completed within 30 days of the end of the project. It should then be circulated for comment and recommendations to key persons within the monitoring program who are knowledgeable in this area. The report should also be forwarded to the Bureau for filing. A final decision as to instrument purchase should be made within two weeks. The decision will be made at a conference (phone or personal) with representatives of each monitoring group and the QA Coordinator.

NOTE:

When a decision on instrument purchase must be made rapidly, a shortened version of this report - just containing the charts and tables from the evaluations should be circulated for comment immediately. The remainder of the report is still to be written and added to the tables at a later date.

A sample copy of a procurement report is available from the Quality Assurance Coordinator.

FIGURE 1
AMBIENT AIR ANALYZERS
USER FACT SHEET

INSTRUMENT MANUFACTURER AND MODEL NUMBER ________________________

COMPANY NAME ________________________ DATE ________________

COMPANY CONTACT ______________________ PHONE NUMBER __________

DNR CONTACT ________________________

1. GENERAL INFORMATION

   a. How many analyzers do you own?

   b. How long have you operated the analyzers?

2. MECHANICAL DEPENDABILITY

   a. In the time since you have owned the instruments, how many mechanical breakdowns, on average, have you experienced?

   b. Do any specific parts give more breakdown problems than others? If so, which ones?

3. ELECTRONIC DEPENDABILITY

a. In the time since you have owned the instruments, how many electronic breakdowns, on average, have you experienced?

b. Do any specific parts give more breakdown problems than others? If so, which ones?

4. CHEMICAL DEPENDABILITY

a. What is the average zero and span drift you see on the instrument - in ppm/x days or in % chart/x days?

b. How frequently do you perform zero/span checks?

c. How frequently do you have to perform a multipoint calibration on the analyzer?

d. How long does it take to perform the multipoint calibration?

e. What is the response time on the instrument - in minutes or seconds - to reach 95% of scale from the baseline?

5. EASE OF WORKING WITH THE INSTRUMENT

a. Are the control switches and knobs easily accessible to the operator?

b. How easy is it to

1. replace boards?

2. clean the instrument?

3. replace filters or other parts?

Be specific (example: flow controllers cannot be reached without dismantling...etc.).

c. How sophisticated must the user be to operate the instrument? Can an engineer operate it? An electronics technician? A chemist?

6. VENDOR RESPONSIVENESS

a. What is the turnaround time on vendor repair of instruments? Be specific - in days, weeks, etc.

b. What is the quality of the repair work performed by the vendor or manufacturer? Be specific.

c. How long does it take to get a vendor or manufacturer repair person to the field?

d. Are vendor representatives knowledgeable about the instrument, its operation and potential problems?

7. COST OF OPERATION

a. What parts, chemicals or other equipment must be replaced frequently?

b. How expensive are replacement parts? Be specific.

c. How much time must be spent by repair people, operators, electronics people, chemists, etc., to keep the instruments operational?

8. INSTRUMENT DOWN TIME

a. What percent data capture do you average, or how many hours/unit time (day, month, etc.) are the instruments inoperable?

b. What is the major reason for your instrument down time?

9. INTERFERENCES

a. Are there any common interferences (gases in the ambient air, at the site; temperature variations, etc.) which affect the response of the instrument? If yes, what are they?

b. How badly is the instrument affected by the interferents? Be specific.

10. GENERAL INFORMATION

a. Have you used any other manufacturer's analyzers? If yes, which ones?

b. If you had a choice, would you purchase this analyzer again? Why?

c. Any other comments not covered above.

FIGURE 2
AMBIENT AIR ANALYZERS
INSTRUMENT PERFORMANCE DATA SHEET

Instrument Manufacturer and Model # ______________________ Date ______________

Instrument Serial # ______________________ Pollutant ______________

EPA PERFORMANCE SPECIFICATIONS		PERFORMANCE SPECIFICATION RESULTS	
Range		(Minimum)	
		(Maximum)	
Noise		(So)	
		(S_{MV})	
Lower Detectable Limit		(B_L-B_Z)	
		(2x noise)	
Interferent Equivalent		(Interferents(s))	
		Result	
	24 hrs.		24 hrs 48 hrs 72 hrs
Zero Drift			
			24 hrs 48 hrs 72 hrs
Span Drift		20%URL	
		80%URL	
Lag Time			
Rise Time			
Fall Time			
Precision		P_{20}	
		P_{80}	

FIGURE 3

PERFORMANCE SPAN AND FLOW RATE CHECKS

Instrument Manufacturer
and Model Number ____________________

Analyzer S/N# ____________________

Calibrator S/N# ____________________

Last Generator Calibration ____________________

Date	Rotameter Setting	Flow cc/min	Calibration Output	Analyzer Response	Sample Flow Rate	*% Diff.

$$\text{*\% Diff.} = \left(\frac{\text{Analyzer Response} - \text{Calibration Output}}{\text{Calibration Output}}\right) 100\%$$

Recorder Zero Setting ____________________

Zero Setting ____________________

Span Setting ____________________

Auxiliary Gas Flow Rate ____________________
(Specify gas)

Method(s) of Analyzer Data Retrieval
Strip Chart ____
Data Averages ____
Data Logger ____

How was sample flow rate determined?

Remarks:

CALIBRATION FORM

1. Instrument manufacturer and model number - The manufacturer's name, the model number of the analyzer.

2. Instrument number - The manufacturer's serial number affixed to the analyzer.

3. Initial calibration date - Date of the first calibration of the analyzer.

   By - Name of person performing the calibration.

4. Final calibration date - The date of the final calibration of the analyzer.

   By - The last name or initials of the person performing the calibration.

5. Generator number - The DNR serial number for the generator used to calibrate the analyzer.

6. Date of last generator calibration - The date the generator was calibrated.

7. Remarks - Any comments on instrument performance that would affect the interpretation of the calibration data.

8. This information is completed for all analyzers.

   a. Sample flow rate - The sample air rotameter setting.

   b. Auxiliary gas flow rate - Flow rate of any gases used by the analyzer (example: air, hydrogen, ethylene).

   c. Zero setting - If the instrument is so equipped, this is the reading on the zero control setting.

   d. Zero offset - The % chart reading when zero volts is applied to the recorder.

   e. Span setting - The reading from the span setting.

9. Number 8 is completed twice for each instrument calibration. The values for each parameter are entered into the INITIAL column when the instrument is first calibrated, and each parameter is then recorded in the FINAL column when the final calibration is completed.

There are eight columns on the bottom half of the form. These are completed for both INITIAL and FINAL calibrations.

1. Generator setting - In this column place first the number on the lamp position switch if ozone is used. After it, place the rotameter setting.

2. Generator concentration ppm - The output from the generator for the rotameter setting and position switch setting used.

3. Initial chart reading % FS - This column lists the % chart reading for the pollutant value in column 2 when the instrument has the span setting found in the INITIAL column.

4. Initial instrument reading ppm - This is the instrument reading found on the instrument panel when a given quantity of pollutant is passed to it.

5. Final chart reading % FS - This column lists the % chart reading for the pollutant value in column 2 when the instrument has the span setting found in the FINAL column.

6. Final instrument reading ppm - This is the instrument reading for a given quantity of pollutant at the FINAL calibration.

7. Percent - This is the percent deviation of the instrument from the true pollutant concentration. It is calculated twice. The top half of the column gives the initial percent. This is defined as:

   $$\frac{\text{Generator ppm} - \text{Initial reading}}{\text{Generator ppm}} \times 100$$

   or

   $$\frac{\text{Column 4} - \text{Column 2}}{\text{Column 2}} \times 100$$

   The bottom half of the column lists the final percent. This is defined as:

   $$\frac{\text{Generator ppm} - \text{Final reading}}{\text{Generator ppm}} \times 100$$

   or

   $$\frac{\text{Column 6} - \text{Column 2}}{\text{Column 2}} \times 100$$

FIGURE 4

CALIBRATION FORM

INSTRUMENT MANUFACTURER AND MODEL NUMBER ____________________ INSTRUMENT NUMBER __________

INITIAL CALIBRATION DATE ____________________ BY __________

FINAL CALIBRATION DATE ____________________ BY __________

GENERATOR NUMBER __________ DATE OF LAST GENERATOR CALIBRATION __________

REMARKS:

	INITIAL CALIB.	FINAL CALIB.
SAMPLE FLOW RATE		
AUXILIARY GAS FLOW RATE (SPECIFY GAS)		
ZERO SETTING		
ZERO OFFSET (% CHART)		
SPAN SETTING		
OTHER (SPECIFY)		

GENERATOR SETTING POSITION SWITCH / ROTO SET. / FLOW (CC/MIN) (1)	GENERATOR POLLUTANT PPM (2)	INITIAL CHART READING (% FS) (3)	INITIAL INSTRU. READING ppb (4)	FINAL CHART READING (% FS) (5)	FINAL INSTRU. READING ppb (6)	Δ % INITIAL / Δ % FINAL	COMMENTS

$$\Delta \% \text{ INITIAL} = \frac{\text{Col. 4} - \text{Col. 2}}{\text{Col. 2}}$$

$$\Delta \% \text{ FINAL} = \frac{\text{Col. 6} - \text{Col. 2}}{\text{Col. 2}}$$

FIGURE 5

CALIBRATION DRIFT TEST

INITIAL SLOPE ____________________ FINAL SLOPE ____________________

INITIAL INTERCEPT ____________________ FINAL INTERCEPT ____________________

DATE OF CALIBRATION ____________________ DATE OF CALIBRATION ____________________

AVG. % DIFFERENCE ____________________

% CHART	INITIAL VALUE	FINAL VALUE	% DIFFERENCE
10			
20			
30			
40			
50			
60			
70			
80			
90			
100			

$$\% \text{ DIFF.} = \frac{\text{FINAL VALUE} - \text{INITIAL VALUE}}{\text{INITIAL VALUE}} \times 100$$

APPENDIX 5

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES AND HOLDING TIMES FOR SAMPLE COLLECTION IN THE 106, 208, 404(b)(1) AND THE GREAT LAKES NATIONAL MONITORING PROGRAMS

PARAMETER	PRESERVATIVE	BOTTLE TYPE AND SIZE: SPECIAL INSTRUCTIONS	MAXIMUM HOLDING TIME FROM COLLECTION OF SAMPLE TO START OF ANALYSIS
Microbiology	Ice	300ml sterilized glass or plastic container with 0.2ml of 10% sodium thiosulfate solution, which will neutralize 15mg/l of residual chlorine	8 hours
General Chemistry Acidity Alkalinity Biochemical Oxygen Demand Color Chromium, hexavalent Hardness (EDTA) Nitrogen, Nitrite pH (LAb) phosphorus (ortho) Solids (All Forms) Specific Conductance Sulfite Turbidity MBAS	Ice	960ml oblong polyethylene bottle (Monsanto) with a 43-400mm white polypropylene lineless smooth edge cap. Container is to remain closed until time to remove aliquot for analysis. Orthophosphate sample is filtered at time of collection into a 360ml oblong polyethylene bottle with a 43-400mm white flexidome polypropylene cap.	24 hours for all parameters except turbidity. Turbidity may be held for 7 days prior to analysis. Phosphorus (ortho) analysis on open lake samples shall be started immediately after collection.
Boron Chloride Fluoirde Silica Sulfate	Ice	360ml oblong polyethylene bottle with 38-400mm white flexidome polypropylene cap.	30 days except for open lake silica analysis which shall be initiated immediately after sample collection.

APPENDIX 5 (Continued)

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES AND HOLDING TIMES FOR SAMPLE COLLECTION IN THE 106, 208, 404(b)(1) AND THE GREAT LAKES NATIONAL MONITORING PROGRAMS

PARAMETER	PRESERVATIVE	BOTTLE TYPE AND SIZE: SPECIAL INSTRUCTIONS	MAXIMUM HOLDING TIME FROM COLLECTION OF SAMPLE TO START OF ANALYSIS
Non-halogenated Organic Compounds	For compounds other than the halogenated pesticides, preservative is to be coordinated with the Chief, QAO, to meet the needs of the proposed survey.	One quart glass bottle with teflon cap liner. The number of sample aliquots necessary for specific organic compounds analysis will be determined prior to sampling after consultation with the QAO.	24 hours
Chlorinated hydrocarbons	None	One liter glass with teflon lined cap.	30 days
Nutrients, Total Carbon, Total Organic (TOC) Chemical Oxygen Demand (COD) Nitrogen, Ammonia Nitrogen, Nitrate/Nitrite Nitrogen, Total Kjeldahl (TKN) Phosphorus (Total)	Sufficient H_2SO_4 to acidify the sample to a pH of less than 2, store at room temperature.	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. Sample aliquots that are composited are to be iced until composite is made and the preservative is added.	30 days for COD/TOC 30 days for TKN 60 days for Total P 30 days for NO_2+NO_3-N 7 days for NH_3-N sewage samples 30 days for NH_3-N surface water samples, except open lake samples, which are to be analysed immediately after collection.
Nutrients, Dissolved Carbon, Dissolved Organic Phosphorus, Dissolved	Sufficient H_2SO_4 to acidify the sample to a pH of less than 2, store at room temperature	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. Filter at time of collection. Filter aliquots that are to be composited to be iced until composite is made and the preservative is added.	30 days for dissolved organic carbon 60 days for dissolved P

APPENDIX 5 (Continued)

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES AND HOLDING TIMES FOR SAMPLE COLLECTION IN THE 106, 208, 404(b)(1) AND THE GREAT LAKES NATIONAL MONITORING PROGRAMS

PARAMETER	PRESERVATIVE	BOTTLE TYPE AND SIZE: SPECIAL INSTRUCTIONS	MAXIMUM HOLDING TIME FROM COLLECTION OF SAMPLE TO START OF ANALYSIS
Oil and Grease	Sufficient H_2SO_4 to acidify the sample to a pH of less than 2, ice.	One quart glass container, with teflon or aluminum foil cap liner	24 hours
Metals, Total Aluminum Arsenic Barium Beryllium Cadmium Calcium Chromium Cobalt Copper Iron Lead Manganese Magnesium Nickel Potassium Selenium Silver Sodium Tin Titanium Vandamium Zinc Hardness (calc)	Sufficient HNO_3 to acidify the sample to a pH of less than 2.	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. Aliquots for composite need not be acidified prior to compositing. Silver is unstable under HNO_3 preservation.	6 months, except for silver. Silver is unstable with any preservative.
Mercury	5ml of .5% HNO_3/L and 10ml of 0.05% $K_2Cr_2O_7$/L	360ml oblong polyethylene bottle with a 38-400 white flexidome polypropylene cap. Aliquots for composite must be acidified at time of sample collection.	13 days
Metals, Dissolved Parameters same as total metals	Sufficient HNO_3 to acidify the sample to pH of less than 2.	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. Filter at time of collection. Acidify immediately after filtration.	6 months, except for silver. Silver is unstable with any preservative.

APPENDIX 5 (Continued)

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES AND HOLDING TIMES FOR SAMPLE COLLECTION IN THE 106, 208, 404(b)(1) AND THE GREAT LAKES NATIONAL MONITORING PROGRAMS

PARAMETER	PRESERVATIVE	BOTTLE TYPE AND SIZE: SPECIAL INSTRUCTIONS	MAXIMUM HOLDING TIME FROM COLLECTION OF SAMPLE TO START OF ANALYSIS
Phenolics	Sufficient H_3PO_4 to acidify sample to pH of less than 2. 1.0 g$CUSO_4$ per/L, ice.	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. If the sample contains oxidizing agents such as chlorine, remove immediately after sample collection by adding an excess of $FeSO_4$ or $NaAsO_2$. Aliquots for composite sample are preserved immediately after collection. (For manual method - 960ml oblong polyethylene bottle with 43-200mm polypropylene lineless smooth edge cap).	14 days
Cyanide	Sufficient NaOH to raise the sample pH to 12, ice	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. Oxidizing agents such as chlorine decompose most of the cyanides. The sample is to be dechlorinated prior to preservation. Dechlorination is accomplished by the addition 0.06g of ascorbic acid per liter of sample. (For manual method - 960ml oblong polyethylene bottle with 43-400mm polypropene lineless smooth edge cap).	7 days
Special Samples Sulfide	Add 4 drops of 2N zinc acetate per 100ml of bottle size. Ice.	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap. Add the preservative to the sample bottle, then fill bottle completely with sample and cap immediately.	30 days.

APPENDIX 5 (Continued)

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES AND HOLDING TIMES FOR SAMPLE COLLECTION IN THE 106, 208, 404(b)(1) AND THE GREAT LAKES NATIONAL MONITORING PROGRAMS

PARAMETER	PRESERVATIVE	BOTTLE TYPE AND SIZE: SPECIAL INSTRUCTIONS	MAXIMUM HOLDING TIME FROM COLLECTION OF SAMPLE TO START OF ANALYSIS
Bottom Sediments Chemistry - Other than Pesticides and PCB's	Ice	360ml oblong polyethylene bottle with a 38-400mm white flexidome polypropylene cap.	30 days
Chemistry PCB's, Pesticides, Phthalate Esters	Ice	One quart glass with mouth, hexane rinsed bottle with teflon cap liner.	30 days
Microbiology	Ice	Whirl-Pak plastic bag.	8 hours
Periphyton Chlorophyll	Freeze	Filter sample through 0.45u membrane filter. Place filter in small bottle (glass or plastic) and freeze immediately. Keep sample in the dark.	6 months
Phytoplankton Chlorophyll	Freeze	Filter sample through 0.45u membrane filter. Place filter in small bottle (glass or plastic) and freeze immediately. Keep sample in the dark. If it is not feasible to filter sample in the field, keep sample in the dark and ice until the sample can reach the laboratory for filtration.	6 months
Macroinvertebrates	70% Ethanol	Appropriate size glass or plastic container.	
Phytoplankton	10ml/L of Lugols sol.	960ml oblong polyethylene bottle (Monsanto) with a 43-400mm white polypropylene lineless smooth edge cap.	6 months

APPENDIX 5 (Continued)

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES AND HOLDING TIMES FOR SAMPLE COLLECTION IN THE 106, 208, 404(b)(1) AND THE GREAT LAKES NATIONAL MONITORING PROGRAMS

PARAMETER	PRESERVATIVE	BOTTLE TYPE AND SIZE: SPECIAL INSTRUCTIONS	MAXIMUM HOLDING TIME FROM COLLECTION OF SAMPLE TO START OF ANALYSIS
Periphyton	3 to 5% formalin	Appropriate size glass or plastic container.	6 months
Zooplankton	5% formalin	360ml oblong polyethylene bottle with a 38-400mm white flexidome cap.	6 months

APPENDIX 6

EPA OFFICIAL ANALYTICAL METHODOLOGY

PRIOROTY POLLUTANT MEASUREMENTS

Recommended analytical methods for priority pollutants are described in "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants" available from the Environmental Monitoring and Support Laboratory, EPA, Cincinnati, Ohio 45268.

These guidelines for sampling and analysis of industrial wastes have been prepared by the staff of the Environmental Monitoring and Support Laboratory - Cincinnati, at the request of the Effluent Guidelines Division, Office of Water and Hazardous Wastes, and with the cooperation of the Environmental Research Laboratory, Athens, Georgia. The procedures represent the current state of the art, but improvements are anticipated as more experience with a wide variety of industrial wastes is obtained. Users of these methods are encouraged to identify problems encountered and to assist in updating the test procedures by contacting the Environmental Monitoring and Support Laboratory, EPA, Cincinnati, Ohio 45268. These methods were first made available in March 1977 and were revised in April 1977.

APPENDIX 7

EPA OFFICIAL ANALYTICAL METHODOLOGY

HAZARDOUS WASTE MEASUREMENTS

Samples will be collected in containers prepared by the CRL and shipped to the National Field Investigation Center - Denver, for extraction. The extract will be returned to the CRL lab for analysis.

NEIC expects to be ready to start processing samples in about three months. A safety manual for handling these materials which will presumably contain information on containers and shipping is also being prepared.

The collection of samples, preparation of containers, etc., is to be coordinated through the Director of the CRL. Existing Agency test procedures are to be used until test procedures specifically for the hazardous waste program have been finalized by the Agency.

APPENDIX 8

SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

AMBIENT AIR SAMPLES

PARAMETER	RECOMMENDED HOLDING TIME	PRESERVATION METHOD
Particulate Filters	Indefinite	Store in controlled atmosphere of <50% relative humidity
Sulfur Dioxide (Pararosaniline Method)	30 days, if properly stored	Store at <4°C after collection, during transport, and before analysis
Nitrogen Oxides (Sodium-Arsenite Method)	6 weeks	Samples are stable for 6 weeks at room temperature
Fluoride	None	Collect and store in plastic containers

APPENDIX 9

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

PARAMETER	METHOD	REFERENCE*
Acidity, as $CaCO_3$, mg/l	Electrometric end point (pH of 8.2) or phenolphthlanein end point	1, 2, 3, 4
Alkalinity, as $CaCO_3$, mg/l	Electrometric titration (only to pH 4.5) manual or automated, or equivalent automated methods	1, 2, 3, 4
Ammonia (as N), mg/l	Manual distillation (at pH 9.5) followed by nesslerization, titration electrode, Automated phenolate	1, 2, 3, 4
Bacteria		
Coliform (fecal), no./100 ml	MPN; membrane filter	2, 4
Coliform (total), no./100 ml	MPN; membrane filter	2, 4
Fecal streptococci, no./100 ml	MPN; membrane filter, plate count	2, 4
Benzidine, mg/l	Oxidation--colorimetric	5
Biochemical oxygen demand 5-d (BOD_5), mg/l	Winkler (Azide modification) or electrode method	2, 4
Bromide, mg/l	Titrimetric, iodine-iodate	1, 3, 4
Chemical oxygen demand (COD), mg/l	Dichromate reflux	1, 2, 3, 4
Chloride, mg/l	Silver nitrate; mercuric nitrate; or automated colorimetric-ferricyanide	1, 2, 3, 4
Chlorinated organic compounds (except pesticides), mg/l	Gas chromatography	6
Chlorine--total residual, mg/l	Iodometric titration, amperometric or starch-iodine end-point; DPD colorimetric or Titrimetric methods (these last 2 are interim methods pending laboratory testing)	1, 2, 3
Color, platinum cobalt units or dominant wavelength, hue luminance, purity	Colorimetric; spectrophotometric; or ADMI procedure	1, 2, 4

APPENDIX 9 (Continued)

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

PARAMETER	METHOD	REFERENCE*
Cyanide, total, mg/l	Distillation followed by silver nitrate titration or pyridine pyrazolone (or barbituric acid) colorimetric	1, 2, 3, 4
Dissolved oxygen, mg/l	Winkler (Azide modification) or electrode method	1, 2, 3, 4
Fluoride, mg/l	Distillation followed by ion electrode; SPADNS; or automated complexone	1, 2, 3, 4
Hardness--total, as $CaCO_3$, mg/l	EDTA titration; automated colorimetric; or atomic absorption (sum of Ca and Mg as their respective carbonates)	1, 2, 3, 4
Hydrogen ion (pH), pH units	Electrometric measurement	1, 2, 3, 4
Kjeldahl nitrogen (as N), mg/l	Digestion and distillation followed by nesslerization, titration, or electrode; automated digestion automated phenolate	1, 2, 4
Metals		
All dissolved metals	0.45 micron filtration[7] followed by referenced method for total metal	1, 2, 4
Aluminum--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Eriochrome Cyanine R)	1, 2, 4
Antimony--total, mg/l	Digestion[8] followed by atomic absorption[9]	1
Arsenic--total, mg/l	Digestion followed by silver diethyldithiocarbamate; or atomic absorption[9]	1, 2, 4
Barium--total, mg/l	Digestion[8] followed by atomic absorption[9]	1, 2, 4
Beryllium--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Aluminon)	1, 2, 4
Boron--total, mg/l	Corimetric (Curcumin)	1, 2

APPENDIX 9 (Continued)

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

PARAMETER	METHOD	REFERENCE*
Cadmium--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Dithizone)	1, 2, 3, 4
Calcium--total, mg/l	Digestion[8] followed by atomic absorption; or EDTA titration	1, 2, 3, 4
Chromium VI, mg/l	Extraction and atomic absorption; colorimetric (Diphenylcarbazide)	1, 2, 4
Chromium--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Diphenylcarbazide)	1, 2, 3, 4
Cobalt--total, mg/l	Digestion[8] followed by atomic absorption[9]	1, 2, 3, 4
Copper--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Neocuproine)	1, 2, 3, 4
Gold--total, mg/l	Digestion[8] followed at atomic absorption[9]	
Iridium--total, mg/l	Digestion[8] followed by atomic absorption[9]	
Iron--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Phenanthroline)	1, 2, 3, 4
Lead--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Dithizone)	1, 2, 3, 4
Magnesium--total, mg/l	Digestion[8] followed by atomic absorption[9] or gravimetric	1, 2, 3, 4
Manganese--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Persulfate or periodate)	1, 2, 3, 4
Mercury--total, mg/l	Flameless atomic absorption	1, 2, 3, 4
Molybdenum--total, mg/l	Digestion[8] followed by atomic absorption[9]	1, 3
Nickel--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Heptoxime)	1, 2, 3, 4
Osmium--total, mg/l	Digestion[8] followed by atomic absorption[9]	

APPENDIX 9 (Continued)

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

PARAMETER	METHOD	REFERENCE*
Palladium--total, mg/l	Digestion[8] followed by atomic absorption[9]	
Platinum--total, mg/l	Digestion[8] followed by atomic absorption[9]	
Potassium--total, mg/l	Digestion[8] followed by atomic absorption colorimetric (Cobaltinitrite), or by flame photometric	1, 2, 3, 4
Rhodium--total, mg/l	Digestion[8] followed by atomic absorption[9]	
Ruthenium--total, mg/l	Digestion[8] followed by atomic absorption[9]	
Selenium--total, mg/l	Digestion[8] followed by atomic absorption[9]	1, 2
Silica--dissolved, mg/l	0.45 micron filtration[7] followed by colorimetric (Molybdosilicate)	1, 2, 3, 4
Silver--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Dithizone)	1, 2, 4
Sodium--total, mg/l	Digestion[8] followed by atomic absorption or by flame photometric	1, 2, 3, 4
Thallium--total, mg/l	Digestion[8] followed by atomic absorption[9]	1
Tin--total, mg/l	Digestion[8] followed by atomic absorption[9]	1, 4
Titanium--total, mg/l	Digestion[8] followed by atomic absorption[9]	1
Vanadium--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Gallic acid)	1, 2, 3, 4
Zinc--total, mg/l	Digestion[8] followed by atomic absorption[9] or by colorimetric (Dithizone)	1, 2, 3, 4
Nitrate (as N), mg/l	Cadmium reduction; brucine sulfate; automated cadmium or hydrazine reduction[10]	1, 2, 3, 4
Nitrite (as N), mg/l	Manual or automated colorimetric (Diazotization)	1, 2, 4
Oil and grease, mg/l	Liquid-liquid extraction with trichloro-trifluoroethane-gravimetric	1, 2

APPENDIX 9 (Continued)

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

PARAMETER	METHOD	REFERENCE*
Organic carbon--total (TOC), mg/l	Combustion--infrared method[11]	1, 2, 3
Organic nitrogen (as N), mg/l	Kjeldahl nitrogen minus ammonia nitrogen	1, 2, 4
Orthophosphate (as P), mg/l	Manual or automated ascorbic acid reduction	1, 2, 3, 4
Pentachlorophenol, mg/l	Gas chromatography[6]	2, 3
Pesticides, mg/l	6	
Phenols, mg/l	Distillation followed by colorimetric (4AAP)	1, 2, 3
Phosphorus (elemental), mg/l	Gas chromatography	12
Phosphorus--total (as P), mg/l	Persulfate digestion followed by manual or automated ascorbic acid reduction	1, 2, 3, 4
Radiological		
Alpha--total, pCi per liter	Proportional or scintillation counter	2, 3, 4
Alpha--counting error, pCi per liter		2, 3, 4
Beta--total, pCi per liter	Proportional counter	2, 3, 4
Beta--counting error, pCi per liter		2, 3, 4
Radium--total, pCi per liter		2, 3
Ra, pCi per liter	Scintillation counter	1, 4
Residue		
Total, mg/l	Gravimetric, 103 to 105°C	1, 2
Total dissolved (filterable), mg/l	Glass fiber filtration, 180°C	1, 2
Total suspended (nonfilterable), mg/l	Glass fiber filtration, 103 to 105°C	1,2
Settleable, ml/l or mg/l	Volumetric or gravimetric	2
Total volatile, mg/l	Gravimetric, 550°C	1, 2
Specific conductance, micromhos per centimeter at 25°C	Wheatstone bridge conductimetry	1, 2, 3, 4

APPENDIX 9 (Continued)

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

PARAMETER	METHOD	REFERENCE*
Sulfate (as SO_4), mg/l	Gravimetric; turbidimetric; or automated colorimetric (barium chloranilate)	2, 3
Sulfide (as S), mg/l	Titrimetric--iodine for levels greater than 1 mg per liter; Methylene blue photometric	1, 3, 4
Sulfite (as SO_3), mg/l	Titrimetric, iodine-iodate	1, 2, 3
Surfactants, mg/l	Colorimetric (Methylene blue)	1, 2, 3, 4
Temperature, °C	Calibrated glass or electrometric thermometer	1, 2, 4
Turbidity, NTU	Nephelometric	1, 2, 3, 4

*References:

[1] Methods for Chemical Analysis of Water and Wastes, 1979. U.S. Environmental Protection Agency. Office of Research and Development. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

[2] Standard Methods for the Examination of Water and Wastewater, 14th edition, 1975. American Public Health Association, American Water Works Association and Water Pollution Control Federation, Washington, D.C.

[3] Annual Book of ASTM Standards, Part 31: Water, 1978. American Society for Testing and Materials, Philadelphia, Pennsylvania.

[4] All references for USGC methods, unless otherwise noted, are to Brown, E., Skougstad, M.W., and Fishman, M.J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases". U.S. Geological Survey Techniques of Water-Resources Inv., Book 5, Ch. A1 (1970).

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[5] Adequately tested methods for benzidine are not available. Until approved methods are available, the following interim method can be used for the estimation of benzidine: (1) "Method for Benzidine and Its Salts in Wastewaters," available from Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[6] Procedures for pentachlorophenol, chlorinated organic compounds, and pesticides can be obtained from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[7] Dissolved metals are defined as those constituents which will pass through a 0.45 um membrane filter. A prefiltration is permissible to free the sample from larger suspended solids. Filter the sample as soon as practical after collection using the first 50 to 100 ml to rinse the filter flask. (Glass or plastic filtering apparatus are recommended to avoid possible contamination). Discard the portion used to rinse the flask and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO_3 to a pH of 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the samples.

[8] For the determination of total metals the samples is not filtered before processing. Because vigorous digestion procedures may result in a loss of certain metals through precipitation, a less vigorous treatment is recommended as given on page 83 (4.4.4) of "Methods for Chemical Analysis of Water and Wastes" (1974). In those instances where a more vigorous digestion is desired, the procedure on page 82 (4.1.3) should be followed. For the measurement of the noble metal series (gold, iridium, osmium, palladium, platinum, rhodium, and ruthenium), an aqua regia digestion is to be substituted as follows: Transfer a representative aliquot of the well-mixed sample to a Griffin beaker and add 3 ml of concentrated redistilled HNO_3. Place the beaker on a steam bath and evaporate to dryness. Cool the beaker and cautiously add a 5 ml portion of aqua regia. (Aqua regia is prepared immediately before use by carefully adding 3 volumes of concentrated HCl to one volume of concentrated HNO_3). Cover the beaker with a watch glass and return to the steam bath. Continue heating the covered beaker for 50 min. Remove cover and evaporate to dryness. Cool and take up the residue in a small quantity of 1:1 HCl. Wash down the beaker walls and watch glass with distilled water and filter the sample to remove silicates and other insoluble material that could clog the atomizer. Adjust the volume to some predetermined value based on the expected metal concentration. The sample is now ready for analysis.

APPENDIX 9 (CONTINUED)

EPA OFFICIAL ANALYTICAL METHODOLOGY

WATER QUALITY MEASUREMENTS

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[9] As the various furnace devices (flameless AA) are essentially atomic absorption techniques, they are considered to be approved test methods. Methods of standard addition are to be followed as noted in page 78 of "Methods for Chemical Analysis of Water and Wastes", 1974.

[10] An automated hydrazine reduction method is available from the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

[11] Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water": U.S. Geological Survey Techniques of Water-Resources Inv., Book 5, Ch. A3 (1972).

[12] R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", "Journal of Chromatography", Vol. 47, No. 3, pp. 421-426, 1970.

APPENDIX 10

EPA OFFICIAL ANALYTICAL METHODOLOGY

RADIATION METHODS

PARAMETER AND UNITS	METHOD	SAMPLE MATRIX	REFERENCE
Alpha - total pCi per liter	Proportional or scintillation counter	Water	Interim Radiochemical Methodology for Drinking Water EPA-600/4-75-008 Standard Methods for the Examination of Water and Wastewater, 14th Ed.
Beta - total pCi per liter	Proportional counter	Water	(same as above)
Radium-226 - pCi per liter	Scintillation counter	Water	(same as above)
Strontium 89, 90 - pCi per liter		Water	(same as above)
Tritium - pCi per liter		Water	(same as above)
Cesium 134 - pCi per liter		Water	(same as above) ASTM D-2459 Gamma Spectroscopy in Water, 1975
Uranium - pCi per liter	Fluorometric	Water	ASTM D-2907 Micro Quantities of Uranium in Water by Fluorimetry, 1975
Others (varium units depending on media)		Various	HASL Procedure Manua, HASL 300, ERDA Health and Safety Laboratory, New York, NY, 1973

APPENDIX 11

EPA OFFICIAL ANALYTICAL METHODOLOGY

AMBIENT AIR MEASUREMENTS

POLLUTANT	MEASUREMENT METHOD OR PRINCIPLE	REFERENCE
Suspended Particulates	High Volume sampler Tape sampler	CFR 40, Part 50, Appendix B, July 1, 1979
Sulfur Dioxide	Pararosaniline or equivalent	CFR 40, Part 50, Appendix A, July 1, 1979
Carbon Monoxide	Nondispersive infrared or equivalent	CFR 40, Part 50, Appendix C, July 1, 1979
Photochemical Oxidants	Gas phase chemiluminescence or equivalent	CFR 40, Part 50, Appendix D, July 1, 1979
Nitrogen Dioxide	Gas phase chemiluminescence or equivalent	CFR 40, Part 50, Appendix F, July 1, 1979

APPENDIX 12

EPA OFFICIAL ANALYTICAL METHODOLOGY

SOURCE AIR MEASUREMENTS

DETERMINATION	DESCRIPTION OF METHOD	REFERENCE
Sample and Volocity Traverses		EPA Method 1 Environmental Protection Agency Performance Test Methods, page I-1, EPA-340/1-78-011
Stack Gas Velocity	Pitot	EPA Method 2 I-13
Dry Molecular Weight of Gas	Orsat	EPA Method 3 I-45
Stack Gas Moisture	Volumetric & Gravimetric	EPA Method 4 I-61
Particulate Emissions	Gravimetric	EPA Method 5 I-79
Sulfur Dioxide	Collection by impinger, analysis by barium perchlorate titration	EPA Method 6 I-119
Nitrogen Oxide	Collection by evacuated flask, colorimetric analysis	EPA Method 7 I-135
Sulfuric Acid Mist and Sulfur Dioxide	Collection by impinger, analysis by barium perchlorate titration	EPA Method 8 I-157
Visible Emissions	Certified observer	EPA Method 9
Carbon Monoxide	Non-dispersive infrared	EPA Method 10
Hydrogen Sulfide	Collection by impinger, iodimetric titration	EPA Method 11

APPENDIX 12 (CONTINUED)

EPA OFFICIAL ANALYTICAL METHODOLOGY

SOURCE AIR MEASUREMENTS

DETERMINATION	DESCRIPTION OF METHOD	REFERENCE
Fluoride	Collection by impinger, colorimetric, or specific ion electrode	EPA Method 13A (Colorimetric) or 13B (Specific Ion Electrode)
Sulfur Compounds	Gas chromatographic determination of sulfur gases emitted by a Claus Sulfur Recovery Unit	EPA Method 15
Sulfur Compounds	Gas chromatographic determiantion of reduced sulfur compounds emitted by paper mills	EPA Method 16
Particulate Matter	In-stack filter determination of particulate matter	EPA Method 17

APPENDIX 13

EPA OFFICIAL ANALYTICAL METHODOLOGY

PUBLIC WATER SUPPLY METHODS

Parameter	Method	Reference
Organics (a) Chlorinated Hydrocarbons: Endrin Lindane Methoxychlor Toxaphene	Gas chromatography with electron capture detector	Methods for Organochlorine Pesticides in Industrial Effluents, MDQARL, EPA, Cincinnati, Ohio, 1973
(b) Chlorophenoxys: 2,4-Dichlorophenoxyacetic acid 2,4,5-Trichlorophenoxypropionic acid		Methods for Chlorinated Phenoxy Acid Herbicides in Industrial Effluents, MDQARL, EPA, Cincinnati, Ohio, 1973
Radiation		Code of Federal Regulations, 40 (Parts 100 to 399): 169-197
Inorganic Chemicals		Code of Federal Regulations, 40 (Parts 100 to 399): 169-197
Physical Measurements		Code of Federal Regulations, 40 (Parts 100 to 399): 169-197
Microbiological Measurements		Code of Federal Regulations, 40 (Parts 100 to 399): 169-197

APPENDIX 14

SAMPLE COLLECTION CONTAINERS, PRESERVATIVES, AND HOLDING TIMES FOR SAMPLES COLLECTED IN THE 1412 MONITORING PROGRAM

CHEMISTRY[1]

PARAMETER	PRESERVATIVE[2]	CONTAINER[3]	MAXIMUM HOLDING TIME[4]
Arsenic	Conc. HNO_3 to pH<2	P or G	6 months
Barium	Conc. HNO_3 to pH<2	P or G	6 months
Cadmium	Conc. HNO_3 to pH<2	P or G	6 months
Chromium	Conc. HNO_3 to pH<2	P or G	6 months
Lead	Conc. HNO_3 to pH<2	P or G	6 months
Mercury	Conc. HNO_3 to pH<2	G	38 days
		P	14 days
Nitrate	Conc. H_2SO_4 to pH<2	P or G	14 days
Selenium	Conc. HNO_3 to pH<2	P or G	6 months
Silver	Conc. HNO_3 to pH<2	P or G	6 months
Fluoride	None	P or G	1 month
Chlorinated hydrocarbon	Refrigerate at 4°C as soon as possible after collection	G with foil or Teflon lined cap	14 days[5]
Chlorophenoxys	Refrigerate at 4°C as soon as possible after collection	G with foil or Teflon lined cap	7 days[5]

1 - If a laboratory has no control over these factors, the laboratory director must reject any samples not meeting these criteria and so notify the authority requesting the analyses.

2 - If HNO_3 cannot be used because of shipping restrictions, samples may be initially preserved by icing and immediately shipping it to the laboratory. Upon receipt in the laboratory, the sample must be acidified with concHNO_3 to pH<2. At time of analysis, sample container should be thoroughly rinsed with 1:1 HNO_3; washings should be added to sample.

3 - P = Plastic, hard or solf; G = Glass, hard or soft.

4 - In all cases, samples should be analyzed as soon after collection as possible.

5 - Well stoppered and refrigerated extracts can be held up to 30 days.

APPENDIX 14 (Continued)

RADIOCHEMISTRY[1]

PARAMETER	PRESERVATIVE[2]	CONTAINER[3]
Gross alpha	Conc. HCl or HNO_3 to $pH<2$[4]	P or G
Gross beta	Conc. HCl or HNO_3 to $pH<2$[4]	P or G
Strontium-89	Conc. HCl or HNO_3 to $pH<2$	P or G
Strontium-90	Conc. HCl or HNO_3 to $pH<2$	P or G
Radium-226	Conc. HCl or HNO_3 to $pH<2$	P or G
Radium-228	Conc. HCl or HNO_3 to $pH<2$	P or G
Cesium-134	Conc. HCl to $pH<2$	P or G
Iodine-131	None	P or G
Tritium	None	G
Uranium	Conc. HCl or HNO_3 to $pH<2$	P or G
Photon emitters	Conc. HCl or HNO_3 to $pH<2$	P or G

---

1 - "Federal Register", Volume 41, No. 133, July 9, 1976.

2 - It is recommended that the preservative be added to the sample at the time of collection unless suspended solids activity is to be measured, However, if the sample must be shipped to a laboratory or storage area, acidification of the sample (in its original container) may be delayed for a period not to exceed 5 days. A minimum of 16 hours must elapse between acidification and analysis.

3 - P = Plastic, hard or soft; G = Glass, hard or soft.

4 - If HCl is used to acidity samples which are to be analyzed for gross alpha or gross beta activities, the acid salts must be converted to nitrate salts before transfer of the samples to planchets.

APPENDIX 14 (Continued)

MICROBIOLOGY
DRINKING WATER SAMPLES

Sample bottles must be of at least 120ml capacity, sterile plastic or hard glass, wide mouthed with stopper or plastic screw cap and capable of being sterilized repeatedly. 10mg/l sodium thiosulfate is added to the sample during preparation. Sample volume must be at least 100ml. Samples must be analyzed within 30 hours after collection. If a State principal laboratory is required by State regulations to examine samples after 30 hours and up to 48 hours, the laboratory must indicate that the data may be invalid because of excessive delay before sample analysis. Samples arriving 48 hours shall be refused without exception and a new sample requested.

15-i

APPENDIX 15

SDWA 2-1-10 →

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

H9if

FDM

[redacted]

DATE: MAR 9 1978

JECT: Approved Alternative Analytical Methods - Nationwide Use

ROM: Victor J. Kimm, Deputy Assistant Administrator for Drinking Water (WH-550)

TO: All Regional Administrators

Listed below are additional alternative analytical methods for nationwide use which I have approved for the National Interim Primary Drinking Water Regulations. As stated in my March 10, 1977 memorandum on this subject, publication of new alternate analytical methods will eventually follow in the Federal Register.

Specific questions regarding the details of these procedures should be directed to the Director, Environmental Monitoring and Support Laboratory, Cincinnati.

Measurement	Method
Organics (Pesticides)	"Standard Methods for the Examination of Water and Wastewater," 14th ed., 1975. Organochlorine Pesticides, part 509A, pp. 555-564, Chlorinated Phenoxy Acid Herbicides, part 509B, pp. 565-569.
Fluoride	Modified Automated Alizarin Blue, Ref: "Fluoride in Water and Wastewater," Industrial Method #129-71W, December 1972, Technicon Industrial Systems Tarrytown, NY 10591
Fluoride	Automated Electrode Method, Ref: "Fluoride in Water and Wastewater" Industrial Method #380-75WE, February 2, 1976, Technicon Industrial Systems, Tarrytown, NY 10591

cc: Water
S & A
A & H M

RECEIVED
MAR 15 1978
EPA REGION 5
OFFICE OF REGIONAL ADMINISTRATOR

A Form 1320-6 (Rev. 3-76)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

DATE: SEP 1 1977

SUBJECT: Approved Alternative Analytical Methods - Nationwide Use

FROM: Victor J. Kimm, Deputy Assistant Administrator for Water Supply (WH-550)

TO: All Regional Administrators

RECEIVED
[illegible] 1977
OFFICE OF DIRECTOR
S&A Division, EPA, Region V

This memorandum replaces my earlier memo of May 10 on this subject, since questions at the regional/State level concerning its implementation have been raised. In addition, some points need further clarification and comment prior to official publication of the approved alternate analytical methods in the Federal Register.

In order to expedite the publication of these needed alternate analytical methods and to correct and clarify inaccuracies and other possible ambiguities which may have occurred as the result of collective actions the approved methods for nationwide use are summarized below; hence, my May 10 memo should be disregarded.

Measurement	Method
Arsenic	[1] Flameless Atomic Absorption, Graphite Furnace Technique.
Arsenic	Silver Diethyldithiocarbamate Method, Ref: "Methods for Chemical Analysis of Water and Wastes," pp. 9-10, EPA Office of Technology Transfer, (1974).
Barium	[1] Flameless Atomic Absorption, Graphite Furnace Technique.
Cadmium	[1] Flameless Atomic Absorption, Graphite Furnace Technique.
Chromium	[1] Flameless Atomic Absorption, Graphite Furnace Technique.
Fluoride	Automated Alizarin Fluoride Blue, Ref: "Standard Methods for the Examination of Water and Wastewater," 14, pp. 614-616, (1975)

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Fluoride	[2] Zirconium-Eriochrome Cyanine R, Ref: "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," USGS, Book 5, Chapter A 1, pp. 90-93.
Lead	[1] Flameless Atomic Absorption, Graphite Furnace Technique.
Mercury	Automated Cold Vapor Technique, Ref: "Methods for Chemical Analysis of Water and Wastes," pp. 127-133, EPA Office of Technology Transfer, (1974).
Nitrate	Automated Hydrazine Reduction, Ref: "Methods for Chemical Analysis of Water and Wastes," pp. 185-194, NERC Analytical Quality Control Laboratory, (1971).
Nitrate	Automated Cadmium Reduction, Ref: "Methods for Chemical Analysis of Water and Wastes," pp. 207-212, EPA Office of Technology Transfer, (1974).
Organics[3]	[2] Gas Chromatographic, Ref: "Methods for Analysis of Organic Substances in Water," USGS, Book 5, Chapter A 3, pp. 24-39.
Selenium	[2] Hydride generation - atomic absorption spectrophotometry, USGS. Method, I-1667-77, (1976).
Selenium	Flameless Atomic Absorption, Graphite Furnace Technique, Ref: Atomic Absorption Newsletter, 14, No. 5, pp. 109-116, (1975).
Silver	[1] Flameless Atomic Absorption, Graphite Furnace Technique.

Once it is published, you will be provided with copies of the FR notification by my office. In the interim, these methods may be considered as approved alternative analytical methods to meet the monitoring requirements of the SDWA. Additional information on the flameless atomic absorption graphite furnace technique is available from the Director of the Environmental Monitoring and Support Laboratory in Cincinnati until the 1974 EPA manual is updated.

---

[1] The various furnace devices are considered to be atomic absorption techniques. Methods of standard addition are to be followed as noted on p. 78 of "Methods for Chemical Analysis of Water and Wastes," EPA Office of Technology Transfer, (1974).

[2] Copies available from: Water Quality Branch, National Center U.S. Geological Survey, 112201 Sunrise Valley Drive, Reston, Virginia 22092.

[3] Only the six pesticides named in the Interim Primary Drinking Water Regulations are included: Endrin, Lindane, Methoxychlor, Toxaphene; 2,4-D; and 2,4,5-TP (Silvex). Federal Register, Vol. 40, No. 248, pp. 59570-59571, Dec. 24, 1975.

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APPENDIX 16

PERFORMANCE TESTS FOR THE EVALUATION OF COMPUTERIZED GAS CHROMATOGRAPHY/MASS SPECTROMETRY EQUIPMENT AND LABORATORIES

by

William L. Budde
and
James W. Eichelberger

Environmental Monitoring and Support Laboratory
Office of Research and Development
U.S. Environmental Protection Agency
Cincinnati, Ohio 45268

DISCLAIMER

This report has been reviewed by the Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory - Cincinnati, conducts research to:

+ Develop and evaluate methods to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.

+ Investigate methods for the concentration, recovery, and identification of viruses, bacteria and other microbiological organisms in water; and, to determine the responses of aquatic organisms to water quality.

+ Develop and operate an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.

+ Develop and operate a computerized system for instrument automation leading to improved data collection, analysis, and quality control.

This report was developed by the Advanced Instrumentation Section of the Environmental Monitoring and Support Laboratory. It describes a series of general purpose tests to evaluate the performance of computerized gas chromatography-mass spectrometry (GC/MS) systems. Some of the tests go beyond equipment performance and may be used to evaluate the performance of laboratories using GC/MS for organics analysis. The report will be useful to the many Federal, State, local government, and private laboratories that are planning to employ this powerful analytical tool.

Dwight G. Ballinger
Director
Environmental Monitoring and Support
Laboratory - Cincinnati

ABSTRACT

A series of ten general purpose tests are described which are used to evaluate the performance of computerized gas chromatography-mass spectrometry systems. Evaluation criteria are given with each performance test. Some of the tests go beyond equipment performance, and may be used to evaluate the performance of laboratories using GS/MS for organics analysis.

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The authors wish to acknowledge the careful and competent technical assistance of William Middleton, Jr., who has performed all of the GC/MS tests described in this report at least once, and several of them hundreds of times.

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William Andrade
Region 1
Surveillance and Analysis Division
Lexington, MA 02173

Dwight G. Ballinger, Director
Environmental Monitoring and
Support Laboratory
Cincinnati, OH 45268

Thomas A. Bellar
Environmental Monitoring and
Support Laboratory
Cincinnati, OH 45268

Joseph N. Blazevich
Region 10
Surveillance and Analysis Division
Manchester, WA 98353

Robert L. Booth, Deputy Director
Environmental Monitoring and
Support Laboratory
Cincinnati, OH 45268

Herbert J. Brass
Division of Technical Support
Cincinnati, OH 45268

Aubry E. Dupuy, Jr.
Pesticides Monitoring Laboratory
Bay Saint Louis, MS 39520

Denis Foerst
Environmental Monitoring and
Support Laboratory
Cincinnati, OH 45268

Robert D. Kleopfer
Region 7
Surveillance and Analysis Division
Kansas City, KS 66115

John Kopp
Environmental Monitoring and
Support Laboratory
Cincinnati, OH 45268

John Logsdon
National Enforcement
Investigation Center
Denver, CO 80225

E. William Loy, Jr.
Region 4
Surveillance and Analysis Division
Athens, GA 30601

John M. McGuire
Environmental Research Laboratory
Athens, GA 30601

Aaron A. Rosen
Cincinnati Water Works
Cincinnati, OH 45228

D. C. Shew
R. S. Kerr Environmental
Research Laboratory
Ada, OK 74820

Alan Stevens
Municipal Environmental
Research Laboratory
Cincinnati, OH 45268

Curt Norwood
Environmental Research Laboratory
Narragansett, RI 02882

Dennis R. Seeger
Municipal Environmental
Research Laboratory
Cincinnati, OH 45268

Clois Slocum
Municipal Environmental Research
Laboratory
Cincinnati, OH 45268

Emilio Sturino
Region 5
Surveillance and Analysis Division
Chicago, IL 60606

SECTION 1

INTRODUCTION

This report gives a series of performance tests to evaluate computerized gas chromatography - mass spectrometry (GC/MS) systems. These tests were designed for general use, and are applicable to all types of GC/MS systems. All of the tests use the continuous, repetitive measurement of spectra method of data acquisition, and no selected ion monitoring tests are included. Except for the spectrum validation test (Test I), these performance tests are not intended for routine application in a quality assurance program. Test I is a required daily quality control test for GC/MS systems in routine use for measurements of organic compounds in environmental samples. The other performance tests are intended for use in the evaluation of new GC/MS systems before purchase, or after the completion of the manufacturer's installation. These tests are also useful to evaluate GC/MS performance after a long period of downtime for extensive maintenance or repair, after a long period of equipment neglect or non-use, or as general training experiments for GC/MS operators. Several of the tests go beyond equipment performance and may be used to evaluate the performance of laboratories using GC/MS for organics analysis.

The performance tests described in this report are more rigorous and extensive than the typical manufacturer's installation tests. Indeed, this was intended, and the emphasis of the tests is on an evaluation of the total operating system in a rigorous way using experiments that closely resemble real, day-to-day operating situations. The performance tests should be conducted in the order given, but several are optional or depend on the availability of certain accessories, e.g., the solid probe inlet test.

All the tests described in this report require an operator, and some depend heavily on the skills of laboratory personnel. Therefore, the results of some tests may be limited by the skills available in the laboratory. An experienced, two-person team consisting of a professional scientist and a technician will require approximately three weeks to complete the equipment tests assuming there are no major hardware or software problems. Inexperienced teams or individuals may require anywhere from six weeks to one year to complete all the tests, especially if major hardware or software problems develop. In these tests, the operator and other laboratory personnel are a crucial part of the total operating system.

The examples given in this report reference packed column gas chromatography, but the tests described are equally applicable to open tubular GC/MS systems. With open tubular (capillary) systems some minor adjustments in operating conditions may be necessary.

For all the tests it is assumed that the manufacturer has provided acceptable documentation of users instructions for the operation and maintenance of the GC/MS system. At the very minimum this must include clearly written descriptions of all operating and test functions, clear descriptions of all commands used in the operation of the data system, examples of all commands, and intelligible documentation of error messages. Examples of all outputs must be included as well as error recovery procedures. There must be a narrative description of all data system files, and the narrative should describe the exact nature of the algorithm used for all the significant mass spectrometric processes. The maintenance manuals must include a complete set of hardware engineering drawings, and maintenance must be described in terms of block diagrams, logic diagrams, flow charts, circuit descriptions, and parts lists.

It is also assumed that the laboratory has provided the GC/MS facility with an appropriate environment including air conditioning and other utilities as required, trained management and operating personnel, needed supplies, essential support equipment, and a reasonable amount of working space which allows access at the sides and rear of the system for maintenance.

Finally, a system logbook must be maintained throughout the evaluation period. This must include an entry for every working day noting the status of the system. This entry must be made even if the system is not used on that day, and signed by the responsible person. The logbook must include a complete record of the number of gas chromatographic injections per day, the number of solid probe samples, all chromatographic column changes, all maintenance procedures, all requirements for service from the manufacturer, and each entry must be signed and dated. This information must be summarized in the performance evaluation report, and the mean numbers of gas chromatographic injections and solid probe samples before ion source maintenance (cleaning) must be reported.

SECTION 2

SUMMARY OF PERFORMANCE TESTS

I. Spectrum Validation Test - Uses decafluorotriphenyl phosphine (DFTPP) to determine whether the system gives a 70 ev electron ionization fragmentation pattern similar to that found in the historical mass spectrometry data base, and the required mass resolution and natural abundance isotope patterns. The spectrum of DFTPP must meet the criteria given in Table 2.

II. System Stability Test - Uses DFTPP to test moderate term (20-28 hours) system stability. The criteria given in Test I must be met.

III. Instrument Detection Limit Test - Uses DFTPP to measure the full and valid spectrum detection limit at a defined and tolerable noise level. At a signal/noise = 5, the required instrument detection limits are 50 nanograms for systems used in the analysis of industrial or municipal wastes, and 30 nanograms for systems used in the analysis for ambient or drinking water.

IV. Saturation Recovery Test - Uses DFTPP and p-bromobiphenyl to simulate a frequently encountered situation with real samples. The spectrum of DFTPP, measured within two minutes after the elution of a 250 fold excess of p-bromobiphenyl, must not contain significant contributions from the ions attributable to p-bromobiphenyl.

V. Precision Test - Uses a variety of typical environmental pollutants to determine precision from filling a syringe to peak integration. The mean relative standard deviation for the compounds used in the test which elute as narrow peaks must be 7% or less using either peak areas in arbitrary units or ratios of peak areas. For broad peaks the mean relative standard deviation must be 13% or less.

VI. Library Search Test - Uses data from Test V to evaluate the speed and completeness of the minicomputer library search algorithm. The mean search time, including background subtraction, must be one minute or less, and all test compounds must be identified as most probable except isomers with very similar spectra should not be counted as incorrect.

VII. Quantitative Analysis with Liquid-Liquid Extraction - Uses a variety of environmental pollutants to measure quantitative accuracy and precision of the total analytical method. The mean of the means of the percentages of the true values observed must be in the 68-132% range with a mean relative standard deviation of 38% or less using either internal or external standards. This test also evaluates laboratory performance.

VIII. Quantitative Analysis with Inert Gas Purge and Trap - Uses a variety of compounds to measure quantitative accuracy and precision of the total analytical method. The mean of the mean method efficiencies must be 70% or more. Chloroform efficiency must exceed 90% and all compounds must exceed 30% efficiency. The spectrum of p-bromofluorobenzene must meet the criteria given in Table 7. The mean of the means of the percentages of the true values observed must be in the range of 90-110% with a mean relative standard deviation of 19% or less using either internal or external standards.

IX. Qualitative Analysis with Real Samples - Uses a real sample to evaluate the ability of the system to deal with real sample matrix effects and interferences. All compounds must be correctly identified except isomers with nearly identical mass spectra should not be counted as incorrect. This test also evaluates laboratory performance.

X. Solid Probe Inlet System Test (Optional) - Uses cholesterol to evaluate the spectrum validity achievable with a solid probe inlet system. The spectrum of cholesterol must meet the criteria given in step three of the test.

SECTION 3

EXPERIMENTAL PROCEDURES

I. Spectrum Validation Test

Correct identifications of organic pollutants from gas chromatography mass spectrometry (GC/MS) data require valid mass spectra of the compounds detected. This is prerequisite to the interpretation of the spectra, i.e., either an empirical search for a match within a collection of authentic spectra or an analysis from the principles of organic ion fragmentation. A properly operating and well tuned GC/MS is required to obtain valid mass spectra.

The purpose of this test is to make a quick check - about 15 minutes - of the performance of the total operating system of a computerized GC/MS. Thus with a minimum expenditure of time, an operator can be reasonably sure that the GC column, the enrichment device, the ion source, the ion separating device, the ion detection device, the signal amplifying circuits, the analog to digital converter, the data reduction system, and the data output system are all functioning properly.

An unsuccessful test requires, of course, the examination of the individual subsystems and correction of the faulty component. Environmental data acquired after a successful systems check are, in a real sense, validated and of far more value than unvalidated data. Environmental data acquired after an unsuccessful test may be worthless and may cause erroneous identifications.

It is recommended that the test be applied at the beginning of a work day on which the system will be used and also anytime there is a suspicion of a malfunction. A mass spectrometer which meets the criteria of this test will, in general, generate mass spectra of organic compounds which are very similar, if not identical, to spectra in collections and textbooks which have been developed over the years with other types of spectrometers. If the performance criteria of this test cannot be met by the user, the system is unacceptable for general purpose environmental measurements.

Procedure:

1. Make up a stock solution of decafluorotriphenylphosphine (DFTPP) at one milligram per milliliter (1000 ppm) concentration in acetone (or a hydrocarbon solvent). The reference compound used

in this test is available from PCR, Inc., P. O. Box 1778, Gainesville, Florida, 32602 and may be named bis (perfluorophenyl) phenylphosphine. This stock solution was shown to be 97+% stable after six months and indications are that it will remain usable for several years. Dilute an aliquot of the stock solution to 10 micrograms per milliliter (10 ppm) concentration in acetone. The very small quantity of material present in very dilute solutions is subject to depreciation due to adsorption on the walls of the glass container, reaction with trace impurities in acetone, etc. Therefore, this solution will be usable only in the short term, perhaps one week.

2. Select a GC column for the tests. Any column that elutes DFTPP in a reasonable time may be used, and several suggested columns are listed in Table 1. Parameters should be adjusted to permit at least four mass scans during elution of the DFTPP. This will permit selection of a spectrum that is reasonably free of abundance distortions due to rapidly changing sample pressure.

3. Set the preamplifier to a suitable sensitivity, set the baseline threshold (zero instrument), and calibrate.

4. Prepare for data acquisition with the following variables:

Mass Range:	40-450 amu
Scan Time:	approximately five seconds
Electron Energy:	70 ev
Electron Multiplier:	Not to exceed that recommended by the supplier for the age of the device.

5. Inject with a syringe 50 nanograms (five microliters) of the dilute standard into the GC column.

6. After the acetone elutes from the column and is pumped or diverted from the system, turn on the ionizer and start scanning.

7. Terminate the run after the DFTPP elutes, turn the ionizer and multiplier off, and plot the total ion current profile.

8. Select a spectrum number on the front side of the GC peak as near the apex as possible, select a background spectrum number immediately preceding the peak, and display the background subtracted spectrum. Some data systems permit spectrum averaging to minimize variations in ion abundance due to rapidly changing sample pressure. This option is acceptable, and may be required for narrow peaks from open tubular columns.

9. The mass spectrum can be output in various ways including a plot of the full spectrum on the plotter or cathode ray tube or a print of the full spectrum on a printer or cathode ray tube.

TABLE 1. SUGGESTED GC COLUMNS AND CONDITIONS

Dimension (Type)	Packing	Flow Rate	Temp.	R. Time
2m x 2 mm ID (Glass)	1.95% QF-1 plus 1.5% OV-17 on 80/100 mesh Gas-Chrom Q	30 ml/min	180	4 min
2m x 2 mm ID (Glass)	3% OV-1 on 80/100 mesh Chromosorb W	30 ml/min	220	5 min
2m x 2 mm ID	5% OV-17 on 80/100 mesh Chromosorb W	30 ml/min	220	5 min
2m x 2 mm ID (Glass)	1% SP2250 on 100/120 mesh Supelcoport	30 ml/min	170	5 min
30m x .25mm ID (Glass)	Wall coated SP 2100	2-5 ml/min	40,240	10 min

The spectrum obtained on the test system must contain ion abundances within limits given for the key ions in Table 2 (1).

If the relative abundances are not within the limits specified, the appropriate adjustments must be made, i.e., resolution, source potentials, calibration of the mass scale, source magnet position, etc. The manufacturer may need to be consulted for assistance in this adjustment. Repeat this test until satisfactory results are obtained.

TABLE 2. DECAFLUOROTRIPHENYLPHOSPHINE KEY IONS AND ION ABUNDANCE CRITERIA.[1]

Mass	Ion Abundance Criteria
51	30-80% of Mass 198
68	Less than 2% of Mass 69
70	Less than 2% of Mass 69
127	30-70% of Mass 198
197	Less than 1% of Mass 198
198	Base Peak, 100% Relative Abundance
199	5-9% of Mass 198
275	10-30% of Mass 198
365	At least 1% of Mass 198
441	Present, but less than Mass 443
442	Greater than 40% of Mass 198
443	17-23% of Mass 442

[1] Criteria for masses 51 and 127 are modifications of previous values (1) based on new interlaboratory data.

II. System Stability Test

The purpose of this test is to evaluate moderate term system stability. Repeat the test described in Section I after 20-28 hours. Do not make any adjustments or recalibration of the system between tests except routine overnight procedures. The abundance criteria in Table 2 must be met. If these criteria are not met, the system is too unstable for routine use and must be repaired.

III. Instrument Detection Limit Test

This test is to determine the smallest quantity of standard test material that can be injected into the GC/MS system that gives an acceptable spectrum meeting the criteria in Table 2, but has a sufficiently low level of background signals to allow correct interpretation of that spectrum if the sample was an unknown. A spectrum of a test compound contaminated with background signals to the extent of about 10% or more of its total ion abundance is considered to be difficult or impossible to interpret correctly. This judgment is somewhat variable because 10% background distributed among a large number of small ions may be acceptable, but a distribution among a few large ions will be unacceptable. Therefore, a signal to noise ratio based on a selection of six ions is used to evaluate the detection limit. This also allows a relatively simple calculation of the ratio.

In a GC/MS system there are a number of potential sources of background signals (chemical noise) including septum bleed, stationary phase bleed, vacuum system background from various physical components, and ion source contamin- ation. Furthermore, all signals are dependent on GC column efficiency, enrichment device efficiency, vacuum system efficiency, ionization efficiency, ion transmission efficiency, and detector gain. Therefore, this test is highly sensitive to the specific system configuration (specific GC column, etc.) and the current condition of that system, e.g., condition of the GC column, extent of contamination in the ion source, extent of contamination of the quadrupole rods if a quadrupole instrument, and condition of the electron multiplier. The state of the system should be documented as part of the records of the instrument detection limit test.

Procedure:

1. Make three dilutions of the stock solution of DFTPP described in Test I. The dilutions should have the concentrations of five micrograms per milliliter, one microgram per milliliter, and one-tenth of a microgram per milliliter.

2. Follow the basic procedures given in Test I and make the following series of injections:

Amount Injected	Volumes and Standards
20 nanograms	4 ul of 5 ug/ml standard
10 nanograms	2 ul of 5 ug/ml standard
5 nanograms	1 ul of 5 ug/ml standard
1 nanogram	1 ul of 1 ug/ml standard
100 picograms	1 ul of 0.1 ug/ml standard

3. List the masses, relative abundances, and/or absolute abundances (intensities) of the background subtracted spectra of DFTPP. Subtract the background spectra as described in Test I. If necessary use an extracted ion current profile to locate the GC peak. Discard all spectra that do not meet the criteria in Table 2. From the remaining spectra discard those that do not display at least six non-DFTPP ions with relative abundances greater than 5%. If additional dilutions or measurements are necessary, do them. Table 3 contains all DFTPP ions over 3% relative abundance and Table 4 contains a group of common background ions.

4. For each of the qualified spectra compute the ratio R as follows:

$$R = \frac{(\text{DFTPP})}{(\text{BACKGD})}$$

where:

(DFTPP) = the summation of the abundances of the ions at masses 127, 255, 275, 441, 442 and 443

(BACKGD) = the summation of the abundances of the six most abundant (but each over 5% relative abundance) non-DFTPP background ions

5. Prepare a plot of R values as a function of amount injected. The instrument detection limit defined in this test is for the complete, valid spectrum with a defined level of acceptable noise. This detection limit is the amount injected that gives an R value of five. If sufficient points are available, a good estimate of the instrument detection limit may be obtained from a first or second order regression on this data.

The rationale for the selection of an R value of five is consistent with the previous statement that background ions should be less than about 10% of the total ion abundance in an interpretable spectrum. The average relative abundance of the six DFTPP ions used to compute R is in the 25-35% range. For an R value of five the average relative abundance of the six background ions will be in the 5-7% range, and it is estimated that all background ions under these conditions will be less than 10% of the total ion abundance.

TABLE 3. IONS OVER 3% RELATIVE ABUNDANCE OBSERVED IN THE 70 ev MASS SPECTRUM OF DFTPP

AMU	INTENSITY	PERCENT OF TOTAL INTENSITY
50.0	8.11	1.11
51.0	34.60	4.74
69.0	32.93	4.51
74.0	3.10	0.42
75.0	4.53	0.62
77.0	34.84	4.77
78.0	3.10	0.42
93.0	3.10	0.42
99.0	3.81	0.52
107.0	10.97	1.50
110.0	20.76	2.84
117.0	6.44	0.88
127.0	37.70	5.16
128.0	3.10	0.42
129.0	12.88	1.76
167.0	4.05	0.55
168.0	4.77	0.65
186.0	13.12	1.79
187.0	3.81	0.52
198.0	100.00	13.69
199.0	7.15	0.98
205.0	5.01	0.68
206.0	20.28	2.77
207.0	4.53	0.62
217.0	5.01	0.68
221.0	4.29	0.58
224.0	11.21	1.53
227.0	3.81	0.52
244.0	8.11	1.11
255.0	49.16	6.73
256.0	7.39	1.01
274.0	4.29	0.58
275.0	23.15	3.17
276.0	3.81	0.52
296.0	5.01	0.68
423.0	3.34	0.45
441.0	9.30	1.27
442.0	69.45	9.51
443.0	12.88	1.76

TABLE 4. COMMON BACKGROUND IONS IN GC/MS SYSTEMS

Masses	Sources
41,43,55,57, 69,71,81,83, 85,95,97,99	Saturated hydrocarbons and unsaturated hydrocarbons - cyclic and open chain-many sources
149	Phthalate esters used as plasticizers in tubing, etc.
73,101,135,197,207 259,345,346,355	Methyl and phenyl silicone polymers used in stationary phases, diffusion pump oil, etc.
169,261	Polyphenyl ether diffusion pump oil

The required instrument detection limits, at an R value of five, are 50 nanograms for systems used in the analyses of industrial or municipal wastes, and 30 nanograms for systems used in analyses of ambient or drinking waters. These limits were obtained from considerations of EPA recommended sample sizes and concentration factors. If a system cannot meet these criteria, maintenance or repair is required. Particular attention should be given to those items mentioned in the second paragraph of this test.

Observed detection limits with this test are as follows:

1. A Finnigan 3200 equipped with a Varian 1400 GC, a packed 1% SP 2250 Column (Table 1), a Systems Industries RIB interface, and a PDP-8 datasystem (disk) gave a detection limit of five nanograms.

2. A Finnigan 4000 with a Finnigan 9610 GC, a packed 1% SP 2250 column (Table 1), an INCOS interface, and an INCOS datasystem (Nova 3, disk) gave a detection limit of 25 nanograms.

IV. Saturation Recovery Test

The purpose of this test is to evaluate the ability of a system to measure the spectrum of a test compound at a low level immediately after a relatively large quantity of another compound entered the system. This situation occurs frequently in real environmental samples, especially waste samples where a very large concentration of one component may saturate the detector, and within a few minutes or less a very small quantity of a compound of interest may enter the detector.

Procedure:

1. Prepare an acetone solution containing five milligrams per milliliter of p-bromobiphenyl and 20 micrograms per milliliter of DFTPP. A second solution containing approximately 50 micrograms per milliliter

of each is optional and may be useful to optimize chromatographic conditions.

2. Establish GC conditions such that the DFTPP elutes within two minutes after the elution of the *p*-bromobiphenyl. These conditions were achieved with a 6' x 2 mm ID glass column packed with 1% SP2250 on Supelcoport (100/120 mesh) using a flow of 30 ml of helium per minute with the initial column temperature at 120°C and programming to 230°C at 10° per minute. The *p*-bromobiphenyl eluted at 110 seconds and the DFTPP at 210 seconds. This test is carried out using the same basic operating parameters given in Test I.

3. Inject two microliters of the standard solution containing the 250:1 ratio of *p*-bromobiphenyl to DFTPP. Plot the DFTPP spectrum as in Test I. Each of the ions at masses 152, 232, and 234, which are the three most abundant in the spectrum of *p*-bromobiphenyl, must be below 5% relative abundance in the background subtracted spectrum of DFTPP.

V. Precision Test

The purpose of this test is to measure the precision of the GC/MS system in quantitative analysis using continuous, repetitive measurement of spectra. This test evaluates precision from filling a syringe to integration of the peak area for a specific quantitation ion. The entire test should be carried out on the same day by the same technician. The application of an automatic sample changer in this test is required if it will be used for normal sample processing. This should be documented in the test results. If acceptable precision cannot be obtained with this test, the precision of a complete anaytical method may also be unacceptable.

Procedure:

1. Select a group of seven or more compounds, and prepare a standard solution in acetone that contains the entire group. Some recommended compounds are in Table 5, and the concentration of each should be 20 micrograms per milliliter. This group of compounds must include a chlorinated hydrocarbon that may decompose on a hot metal surface and a polycyclic aromatic hydrocarbon with a molecular weight greater than 200. For compounds amenable to the inert gas purge and trap procedure, prepare the standard solution in methanol at the same concentration. The purge and trap mixture must include chloroform, bromoform, *sym*-tetrachloroethane, and *p*-bromofluorobenzene. Some recommended compounds are in Tables 9-12. This test may be conducted with either or both groups of compounds.

TABLE 5. PRECISION STATISTICS FOR TEN PRIORITY POLLUTANTS PLUS OCTADECANE

COMPOUND	INTEGRATION MASS	PEAK[1] TYPE	MEAN AREA	S	(S/MEAN AREA) *100
1,3-DICHLOROBENZENE	146	N	6771	278	4.1
NAPHTHALENE	128	N	18077	375	2.1
1,2,4-TRICHLOROBENZENE	180	N	5412	195	3.6
_n_-OCTADECANE	254	N	345	15	4.2
DIMETHYL PHTHALATE	163	N	13540	501	3.7
DI-_n_-BUTYL PHTHALATE	149	N	21770	364	1.7
N-NITROSODIPHENYLAMINE	169	N	6460	228	3.5
HEXACHLOROBENZENE	284	N	4027	139	3.4
PYRENE	202	N	18107	607	3.4
CHRYSENE	228	B	10345	636	6.2
BENZO(A)PYRENE	252	B	9518	681	7.2

[1] N = narrow; B = broad (see text for definitions)

2. Select an appropriate GC column. For compounds similar to those in Table 5, the columns in Table 1 are satisfactory. For compounds, amenable to purge and trap procedures, two acceptable columns are an 8 ft. stainless steel or glass column packed with 1% SP-1000 coated on 60/80 mesh Carbopack B or packed with 0.2% Carbowax 1500 coated on 60/80 mesh Carbopack C. Prepare for data acquisition with the following variables:

   mass range: 35-350 amu (For purge and trap compounds use 20-260 amu)
   scan time: approximately six seconds (two or three seconds with open tubular columns)
   electron energy: 70 ev
   electron multiplier: not to exceed that recommended by the supplier for the age of the device.

3. Inject with a syringe or automatic sample changer four microliters (80 nanograms of each compound) of the standard solution and acquire data until all compounds have eluted from the column. Save the data

file on the data system and repeat the injection a minimum of four times, saving the data files in each case.

4. Plot the total ion current profiles, and use a quantitation program to integrate peak areas in arbitrary units (usually analog-to-digital counts) over a specific quantitation mass for each compound in each data file. Precision may be evaluated using either the peak areas in arbitrary units or ratios of peak areas. The former gives a precision representative of external standardization, and the latter a precision representative of internal standardization. There will be no significant difference in the results using the two methods if the system is operating properly and acceptable syringe filling and injection techniques are used. It is recommended that calculations be carried out using both methods for comparison of results, but the minimum requirement is that precision be evaluated using the method that corresponds to the standardization procedure used in the laboratory for environmental samples.

   Table 5 is an example of data from five replicate syringe injections of 80 nanograms of each compound using a Finnigan 3200 and a PDP-8 based data system. The mean areas are in analog-to-digital converter units and the standard deviations (S) were computed using the equation below. The last column in Table 5 is the relative standard deviation which is (S/mean area)* 100. Table 6 contains the results of computations with exactly the same raw data as in Table 5, but using ratios of areas as in internal standard calibrations. The response factor (RF) is defined in test VII, and the mean response factors are shown in Table 6. The compound di-n-butylphthalate was selected as the internal standard because it showed the smallest variation in peak area (1.7%, Table 5) and eluted near the mid-point in the chromatogram. The standard deviations and relative standard deviations were computed as in Table 5.

$$S = \sqrt{\frac{N \sum_{i=1}^{N} \text{Area}_i^2 - \left(\sum_{i=1}^{N} \text{Area}_i\right)^2}{N\,(N-1)}}$$

where:

S = the standard deviation

N = the number of measurements for each compound

Area = the integrated ion abundance of the quantitation mass

The compounds designated as having narrow peak types in Tables 5 and 6 had widths at half height of 45 seconds or less. The mean relative standard

TABLE 6. PRECISION STATISTICS USING AN INTERNAL STANDARD

COMPOUND	INTEGRATION MASS	PEAK[1] TYPE	MEAN RF	S	(S/MEAN RF) *100
1,3-DICHLOROBENZENE	146	N	0.3112	0.01512	4.9
NAPHTHALENE	128	N	0.83048	0.017250	2.1
1,2,4-TRICHLOROBENZENE	180	N	0.2486	0.008571	3.4
n-OCTADECANE	254	N	0.0158	0.000838	5.3
DIMETHYL PHTHALATE	163	N	0.62202	0.022980	3.7
DI-n-BUTYL PHTHALATE	149	N	1.00000	0.00000	0
N-NITROSODIPHENYLAMINE	169	N	0.2968	0.01008	3.4
HEXACHLOROBENZENE	284	N	0.1850	0.005899	3.2
PYRENE	202	N	0.83171	0.023110	2.8
CHRYSENE	228	B	0.4751	0.02619	5.5
BENZO(A)PYRENE	252	B	0.4370	0.0275	6.3

deviation for the data in Table 5 is 3.3%, and the corresponding mean from Table 6 is 3.6 %. Therefore there was no significant difference in the precision of external and internal standardization. The requirement of this test is that the mean relative standard deviation of data from narrow peaks be 7% or less. This requirement is based on the general observation that data from interlaboratory comparisons is usually about a factor of two more variable than single laboratory data, and this is a reasonable requirement for an acceptable system.

The last two compounds in Tables 5 and 6 gave broader peaks with peak widths at half height of more than 45 seconds. Measurements of these are more variable because of the changing baseline during temperature programming and other factors. The mean relative standard deviations from Tables 5 and 6 are 6.7% and 5.9% respectively, and internal standardization may have some slight advantage for these peaks but there are too few data points to judge the significance of this. The requirement of this test is that the mean relative standard deviation of data from broad peaks be 13% or less. Again the rule of thumb on interlaboratory data was used to establish this requirement.

If this test is conducted with compounds amenable to the inert gas purge and trap procedure, the compound p-bromofluorobenzene must be included in the mixture. This compound is a secondary spectrum validation compound which is used with GC columns that do not elute DFTPP. Therefore, after a purge and trap column is installed for this test p-bromofluorobenzene may be used as a daily check on spectrum validity. The ion abundance criteria for p-bromofluorobenzene are in Table 7, and these are consistent with the DFTPP criteria in Table 2.

TABLE 7. p-BROMOFLUOROBENZENE KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
50	20-40% of the base peak
75	50-70% of the base peak
95	base peak, 100% relative abundance
96	5-9% of the base peak
173	less than 1% of the base peak
174	greater than 50% of the base peak
175	5-9% of mass 174
176	greater than 50% of the base peak
177	5-9% of mass 176

VI. Library Search Test

Minimum requirements for the library search are the availability of the EPA/NIH database which is distributed through the National Bureau of Standards. The searchable database may be a subset of the EPA/NIH database, but the subset must contain at least 10,000 spectra of general and environmental interest and the Chemical Abstracts Service (CAS) registry numbers for each compound. Programs must be available to allow the operator to submit background corrected spectra to the library search, and receive a printed report of the search results. The spectra from one of the experiments in Test V should be submitted to the library search system. Each compound must be identified as the most probable by the library search, except isomers that may have very similar 70 ev EI mass spectra should not be counted as incorrect. The mean search time, including the time for background subtraction, should be one minute or less. Printed reports should include CAS numbers. During this test make several deliberate typical operator errors, such as entry of an incorrect command and a non-existent file name. The data system should respond with an intelligible error message, and return to a logical continuation point.

VII. Quantitative Analysis with Liquid-Liquid Extraction

This test uses a variety of environmental pollutants to measure quantitative accuracy and precision of the total analytical method, but without the complications of real sample matrix effects. The test is designed for laboratories that conduct quantitative analyses of water samples with GC/MS using continuous repetitive measurement of spectra. Therefore, laboratories dealing in other media should design a similar test based on some standard reference material. The principal difference between this test and Test V, the precision test, is the consideration of potential errors and variations due to: (a) extraction of the compounds from a clean water matrix; (b) concentration of the extract to a small volume; and (c) standardization of the measured areas in terms of the concentration of the original sample in micrograms per liter. This is one of the tests that goes beyond equipment performance, and may be used to evaluate the performance of laboratories using GC/MS for organics analysis.

It is recommended that the same standard solution of seven or more compounds that may have been prepared for the precision test (Test V) be used in this test since retention information is already available, and the concentrations are in an acceptable range. However, new standards may be used and the seven or more compounds should be at the 20 microgram per milliliter level in acetone.

Procedure:

1. Add 250 microliters (five micrograms of each compound) of the mixed standard solution in acetone to each of a minimum of five liters of clean water. This aqueous solution is called a laboratory control standard. Set aside one additional liter of clean water as a reagent blank.

2. Carry out the extractions according to the established procedures (2,3,4). The methylene chloride extract must be concentrated to 0.5 milliliter. The blank should be measured first by itself, and if significant contamination is found, correct the problems before proceeding with this test. See the references cited above for information on the interpretation of blanks.

3. Select an appropriate column (Test V), and prepare for data acquisition using the GC/MS operating parameters given in Test V. Inject four microliters of each of the concentrated extracts, and obtain GC/MS data from each injection. Save all of the data files from the minimum of five extracts. Quantitation may be accomplished with either internal or external standardization. If an external standard will be used, this is already prepared and is the solution used to prepare the laboratory control standards. Inject two microliters (40 nanograms) of the external standard and acquire data using the same acquisition parameters.

   If an internal standard will be used, add five microliters of a one milligram per milliliter solution of the internal standard to each of the 0.5 milliliters of concentrated extract. This corresponds to the addition of five micrograms of the internal standard in such a way as to not significantly change the volume of the concentrated extract. Inject four microliters of each extract as above and save all data files. If an internal standard is used it will be necessary to measure the response factors (RF) in a separate experiment with standards (no extraction). The response factors are computed with the following equation:

$$RF = \frac{\dfrac{\text{Area (X)}}{\text{Amount (X)}}}{\dfrac{\text{Area (S)}}{\text{Amount (S)}}}$$

   where: Area(X) = the peak area of the compound in consistent units.

   Amount (X) = the quantity of the compound injected in consistent units.

   Area (S) = the peak area of the internal standard in consistent units.

   Amount (S) = the quantity of internal standard injected in consistent units.

4. Plot the total ion current profiles and use a quantitation program to integrate peak areas in arbitrary units (usually analog-to-digital converter counts) over a specific quantitation mass for each compound in each data file. If an internal standard was employed computations in terms of response factors are acceptable.

5. Precision and accuracy is expressed in terms of the percentages of the true values (P) measured in the experiments and the statistical variations in the data. The standard deviations (S) and the relative standard deviations (S/mean P) *100, are computed as described in Test 5. With an external standard P is computed as follows:

$$P = \frac{\text{area (concentrated extracts)} * 100}{\text{area (external standard)}}$$

With an internal standard P is computed with the equation below which assumes the response factors are defined as above:

$$P = \frac{\text{area (concentrated extract)} \quad * 100}{\text{area (internal standard)} \quad * RF}$$

Table 8 shows precision and accuracy data obtained for eight compounds extracted from clean water with methylene chloride and measured with GC/MS using a single external standard. The GC/MS was a Finnigan model 3200 with a PDP-8 based datasystem. One difference between the data in Table 8 and the procedures described in this test is that the data in Table 8 represents duplicate extractions and measurements at four different concentration levels between 15-200 micrograms per liter for each compound. Figures 1 and 2 show control charts which contain all eight P values for each of two of the compounds. This is a recommended method (5) of displaying precision and accuracy data. Charts should be labelled as in Figures 1 and 2. General experience shows that P values measured over a concentration range of one or two orders of magnitude are often concentration independent within the precision of the method.

The mean of the P values in Table 8 is 84%. Therefore, the requirement of this test is that the mean of the mean P values of the compounds used in this test must be in the range of 68-132%. Again, as in Test V, the expectation is that multi-laboratory data will usually be about a factor of two.more variable than single laboratory data. The mean relative standard deviation from Table 8 is 19%, and the requirement of this test is that the mean relative standard deviation be 38% or less.

VIII. Quantitative Analysis with Inert Gas Purge and Trap

This test uses a variety of environmental pollutants to measure quantitative accuracy and precision of the total analytical method, but without the complications of real sample matrix effects. The test is designed for laboratories that conduct quantitative analyses of water samples with GC/MS using continuous repetitive measurement of spectra. Therefore, laboratories dealing in other media should design a similar test based on some standard reference material. The principal difference between this test and Test V, the precision test, is the consideration of potential errors and variations due to: (a) purging of the compounds from a clean water matrix; (b) trapping and desorption of the compounds; and (c) standardization of the measured areas in terms of the concentration of the original sample in micrograms per liter. This test is required to evaluate purge and trap equipment that is delivered as an integral part of a GC/MS system, or other purge and trap equipment that is interfaced to the GC/MS system.

The series of experiments in this test is used to generate three key pieces of information about purge and trap performance:

TABLE 8. PRECISION AND ACCURACY DATA FOR LIQUID-LIQUID EXTRACTION WITH GC/MS AND AN EXTERNAL STANDARD

COMPOUND	INTEGRATION MASS	MEAN P	S	(S/MEAN P) *100
NITROBENZENE	123	94	8.8	9.4
1,2,3-TRICHLOROBENZENE	180	85	13	15
NAPHTHALENE	128	73	18	25
ACENAPHTHYLENE	152	83	15	18
N-NITROSODIPHENYLAMINE	169	89	19	21
FLUORANTHENE	202	80	19	24
PYRENE	202	83	19	23
n-BUTYLBENZYLPHTHALATE	206	86	17	20

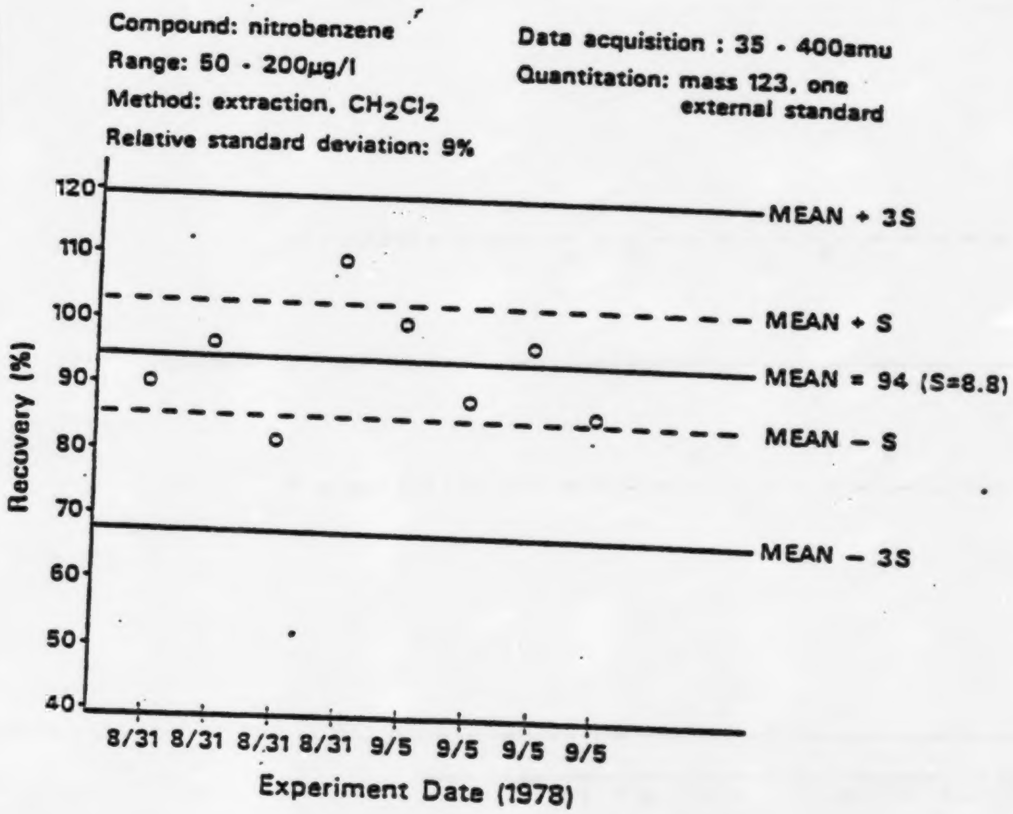


Figure 1. Control chart for nitrobenzene in clean water.

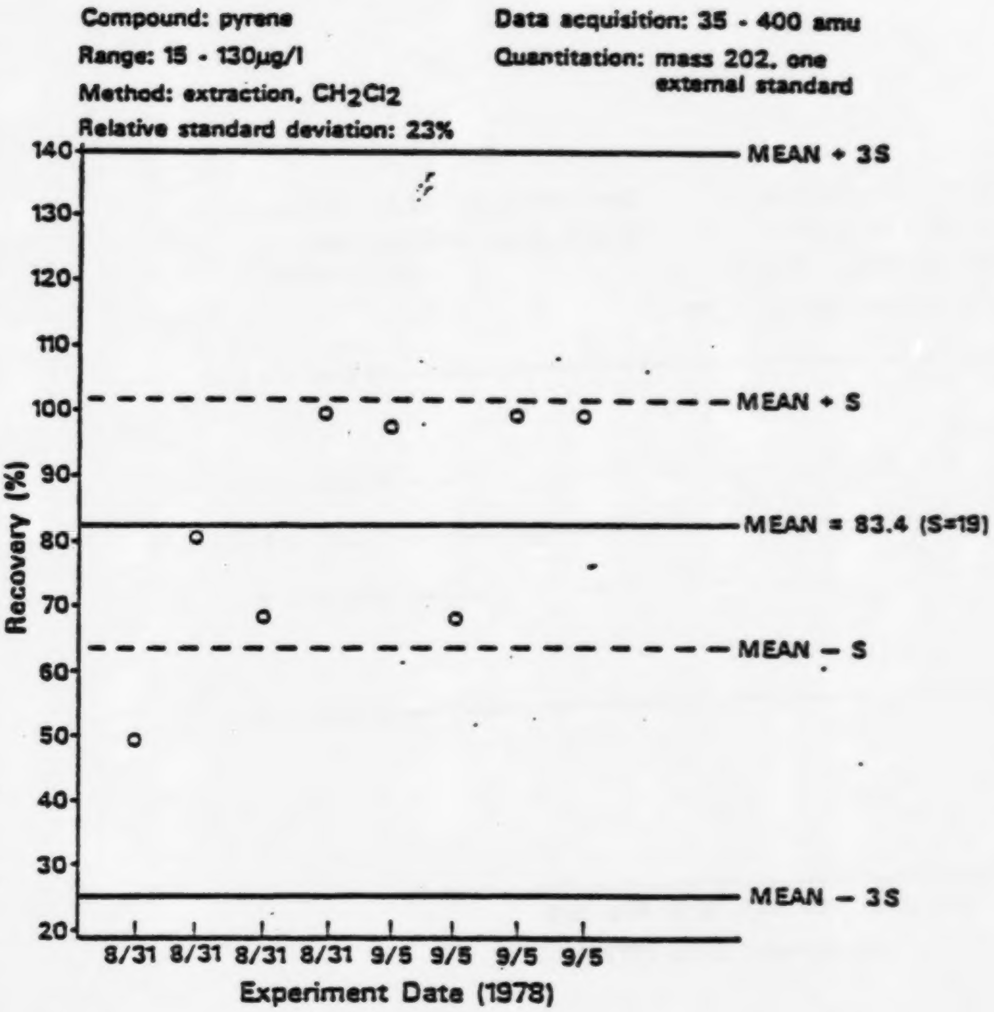


Figure 2. Control chart for pyrene in clean water.

(a) Method efficiency for test compounds by comparison of the measured quantity from syringe injection into the GC with the quantity measured after purging, trapping, and desorption. Because of the method of calibration used in the purge and trap procedure high method efficiency as defined above is not necessary for acceptable precision and accuracy. However, high method efficiency is required for acceptable sensitivity, and low method efficiency will result in unacceptable detection limits. Also in the case of real samples, a low method efficiency combined with an unfavorable matrix effect could render the method totally useless.

(b) Precision of the overall purge, trap, desorption, and GC/MS analysis.

(c) Accuracy of the overall purge, trap, desorption, and GC/MS analysis in terms of the percentage of the true value found in laboratory control standards.

All the above information may be obtained from the same set of data. It is recommended that the same standard solution of seven or more compounds amenable to purge and trap that was recommended for the precision test (Test V) be used in this test since retention information may be already available, and concentrations are in an acceptable range. However, new standards may be used, and the seven or more compounds should be at the 20 micrograms per milliliter level in methanol. The purge and trap mixture must include chloroform, bromoform, sym-tetrachloroethane and p-bromofluorobenzene.

Procedure:

1. Select an appropriate column (see Test V) and prepare for data acquisition using the GC/MS operating parameters given in Test V.

2. Add five microliters (100 nanograms of each compound) of the mixed standard in methanol to each of a minimum of five aliquots of low organic water. Purge and trap samples may be 5 ml to 25 ml, but 5 ml is recommended for optimum method efficiency. This aqueous solution is called a laboratory control standard.

3. Carry out the purge and trap according to the established procedures (2,3,4) at ambient temperature. A low organic water blank should be measured first and at occasional intervals to detect instrument contamination. If significant contamination is found, correct the problems before proceeding with this test. See references cited above for information on the interpretation of blanks.

4. Purge, trap, desorb, and obtain GC/MS data from a minimum of five laboratory control standards and save all the data files. At about the midpoint of the purge and trap analyses, inject with a syringe five microliters (100 nanograms of each compound) of the mixed standard in methanol into the purge and trap GC column. Acquire

GC/MS data using the same acquisition parameters used for purge and trap analyses.

5. Plot the total ion current profiles, and use a quantitation program to integrate peak areas in arbitrary units (usually analog-to-digital converter counts) over a specific quantitation mass for each compound in each data file.

6. Method efficiency must be evaluated by comparing the measured areas from direct GC injection with the corresponding areas from the purge, trap, and desorption experiments. Internal standards cannot be used because method efficiencies for various compounds are not yet known, and comparable response factors cannot be computed for direct injection and purge/trap/desorption.

   Prepare a table similar to Table 9 which shows data obtained with a Finnigan model 3200, a PDP-8 data system, and a Tekmar model, LSC-1 purge and trap device with a 25 ml sample container. The equation used to compute method efficiencies (E) is shown below. The minimum requirement of this test is that the mean of the mean method efficiencies of the compounds used in this test be 70% or more. The chloroform efficiency must exceed 90% and all compounds must be recovered with at least 30% efficiency. Also the spectrum obtained from p-bromofluorobenzene must meet the ion abundance criteria given in Table 7. If these requirements cannot be met, the system is unacceptable for quantitative analyses and needs repair or redesign. One critical method variable that may be optimized is the purge gas flow rate.

$$E = \frac{\text{area (after purge and trap)}}{\text{area (direct injection)}} * 100$$

7. Precision and accuracy data may be obtained by choosing one of the experiments in the purge and trap set as a standard, and computing the percentages of the true values (P) measured in the other laboratory control standards. This is consistent with the standard method of calibration used with the purge and trap method. The experiment chosen as the standard may either be treated as an external standard, or may be used to compute response factors for an internal standard calibration. Table 10 shows the data from the method efficiency determination recomputed by ignoring the direct injection result, and using one of the purge and trap experiments as an external standard. The equation used to compute the percentages of the true values (P) is as follows:

$$P = \frac{\text{area (after purge and trap)}}{\text{area (external standard)}} * 100$$

   The standard deviation of P and relative standard deviation were computed as described in Test V. The mean of the P values in Table 10 is 95% and the mean relative standard deviation is 9.4%. The

TABLE 9. METHOD EFFICIENCIES FOR SOME PRIORITY POLLUTANTS PLUS p-BROMOFLUOROBENZENE

COMPOUND	INTEGRATION MASS	MEAN AREA PURGE/TRAP	AREA DIRECT INJECTION	MEAN METHOD EFFICIENCY(%)
CHLOROFORM	83	2883	3001	96
CARBON TETRACHLORIDE	117	2289	2314	99
BROMODICHLOROMETHANE	83	2925	3280	89
TRICHLOROETHYLENE	130	1474	1653	89
DIBROMOCHLOROMETHANE	129	1572	2343	67
BROMOFORM	173	1241	2788	45
TETRACHLOROETHYLENE	166	1737	2102	83
Sym-TETRACHLOROETHANE	83	1032	3071	34
p-BROMOFLUOROBENZENE	174	1542	2200	70

TABLE 10. PRECISION AND ACCURACY DATA FOR THE PURGE AND TRAP ANALYSIS WITH GC/MS AND AN EXTERNAL STANDARD

COMPOUND	INTEGRATION MASS	MEAN P	S	(S/MEAN P) *100
CHLOROFORM	83	92	8.8	9.5
CARBON TETRACHLORIDE	117	97	7.9	8.2
BROMODICHLOROMETHANE	83	96	7.2	7.5
TRICHLOROETHYLENE	130	94	7.4	7.9
DIBROMOCHLOROMETHANE	129	98	4.4	4.5
BROMOFORM	173	96	5.2	5.4
TETRACHLOROETHYLENE	166	96	14	14
Sym-TETRACHLOROETHANE	83	100	14	14
p-BROMOFLUOROBENZENE	174	90	12	14

requirement of this test is that the mean of the mean P values of the compounds used in this test must be in the range of 90-110%. This is based on the general rule, described in Test V, that data from interlaboratory comparisons is usually about a factor of two more variable than single laboratory data. The mean relative standard deviation must be 19% or less on the same basis.

The percentages of the true values (P) may also be computed by selecting one compound in the test mixture as an internal standard, and using one of the purge and trap experiments to establish response factors as defined in Test VII. The percentages of the true values (P) in the other laboratory control standards are computed as follows (the terms have the same meaning defined in Test VII):

$$P = \frac{\text{area (x)} * 100}{\text{area (s)} * RF}$$

Table 11 shows the method efficiency data recomputed with p-bromofluorobenzene as the internal standard. Response factors were established with the same purge and trap experiment that was used as an external standard for the computations in Table 10. Table 12 shows the same data recomputed with dibromochloromethane as an internal standard. Again, response factors were established with the same purge and trap experiment that was used as an external standard for the computations in Table 10.

The internal standard calculations reveal that the percentages of the true values observed and the relative standard deviations are a function of the internal standard selected. The compound p-bromofluorobenzene eluted late in the chromatogram after temperature programming, and measurements of it were more variable because of this and other factors. This is reflected in the mean of the mean P values from Table 11 of 108% and the mean relative standard deviation of 12%. The compound dibromochloromethane showed the least variation in the external standard data (Table 10) and is an excellent internal standard. The mean of the mean P values from Table 12 is 97% with a mean relative standard deviation of 6.5%. This illustrates that care must be exercised in the selection of an internal standard because of the potentially significant impact on the observed precision and accuracy. The individual P values may also be charted as in Figures 1 and 2 to provide a graphic presentation of the data.

IX. Qualitative Analysis with Real Samples

The purpose of this test is to evaluate the ability of the GC/MS system, laboratory, and sample preparation methods to deal with natural background, interferences, and sample matrices found in real environmental samples. The test is limited to qualitative analyses because of the unpredictable quantitatvie effects of the sample matrix. This is one of the tests that goes beyond equipment performance, and it may be used to evaluate the performance of laboratories using GC/MS for organics analysis. The test is designed for laboratories that conduct qualitative analyses of water samples with GC/MS using continuous, repetitive measurement of spectra.

TABLE 11. PRECISION AND ACCURACY DATA FOR THE PURGE AND TRAP ANALYSIS WITH GC/MS AND THE INTERNAL STANDARD p-BROMOFLUOROBENZENE

COMPOUND	INTEGRATION MASS	MEAN P	S	(S/MEAN P) *100
CHLOROFORM	83	103	13	13
CARBON TETRACHLORIDE	117	108	12	11
BROMODICHLOROMETHANE	83	107	12	11
TRICHLOROETHYLENE	130	105	12	11
DIBROMOCHLOROMETHANE	129	110	11	10
BROMOFORM	173	108	12	11
TETRACHLOROETHYLENE	166	107	13	12
Sym-TETRACHLOROETHANE	83	112	19	17
p-BROMOFLUOROBENZENE	174	100	0	0

TABLE 12. PRECISION AND ACCURACY DATA FOR THE PURGE AND TRAP ANALYSIS WITH GC/MS AND THE INTERNAL STANDARD DIBROMOCHLOROMETHANE

COMPOUND	INTEGRATION MASS	MEAN P	S	(S/MEAN P) *100
CHLOROFORM	83	94	5.8	6.2
CARBON TETRACHLORIDE	117	98	4.3	4.4
BROMODICHLOROMETHANE	83	98	3.7	3.7
TRICHLOROETHYLENE	130	95	3.8	4.0
DIBROMOCHLOROMETHANE	129	100	0	0
BROMOFORM	173	98	2.0	2.0
TETRACHLOROETHYLENE	166	98	9.8	10
Sym-TETRACHLOROETHANE	83	101	11	11
p-BROMOFLUOROBENZENE	174	92	9.7	11

Procedure:

1. Acquire appropriate quality control samples. These should be in sealed glass ampoules containing one to fifty organic compounds dissolved in acetone, methanol, or some other miscible organic solvent. The concentration levels should be suitable for the preparation of aqueous samples in the 10-500 micrograms per liter range by addition of 250 or fewer microliters of the organic solution to 5 to 1000 milliliters of an environmental sample. Instructions for the dilutions must be supplied with the samples, but the identity of the compounds in the ampoules must be supplied separately in sealed envelopes to the laboratory management. Samples of this type are available from:

   John A. Winter, Chief
   Quality Assurance Branch
   EMSL-Cincinnati
   Environmental Protection Agency
   Cincinnati, Ohio 45268

2. Obtain an environmental sample typical of the type normally analyzed in the laboratory. Add the quality control samples to the environmental samples according to the instructions provided, and proceed with the analyses using the appropriate method, e.g., as in Tests VII and VIII.

3. Plot the total ion current profiles and identify all the compounds using the mass spectra. All compounds must be correctly identified except, as in the library search, isomers with nearly identical 70 ev electron ionization spectra should not be counted as incorrect.

X. Solid Probe Inlet System Test (optional)

The purpose of this test is to evaluate the critical thermal characteristics of the solid probe inlet system, and to determine whether valid spectra are produced with this system. The test uses cholesterol which is sensitive to thermal effects. Data acquisition is by continuous repetitive measurement of spectra.

Procedure:

1. Prepare a standard solution of cholesterol in acetone at a concentration of 250 micrograms per milliliter. Evaporate one microliter of this solution in the solid probe sample holder.

2. Use the data acquisition parameters given in Test I, and gradually heat the sample until the cholesterol pressure increases and spectra may be measured.

3. Terminate data acquisition and plot a background subtracted spectrum of cholesterol as described in Test I. Measure the abundances of the ions at masses 386 and 368, and compute the 386/368 abundance

ratio. This should be 3.0 or greater for an acceptable solid probe inlet system. The ion abundance at mass 369 should be 26-34% of the abundance at mass 386. Finally large ions above 30% relative abundance should be at masses 41, 43, 55, 57, 67, 69, 71, 79, 81, 83, 91, 93, 95, 105, 107, 109, 119, 121, 133, 145, 147, 149, 159, 161, 213, 275, 301, and 386.

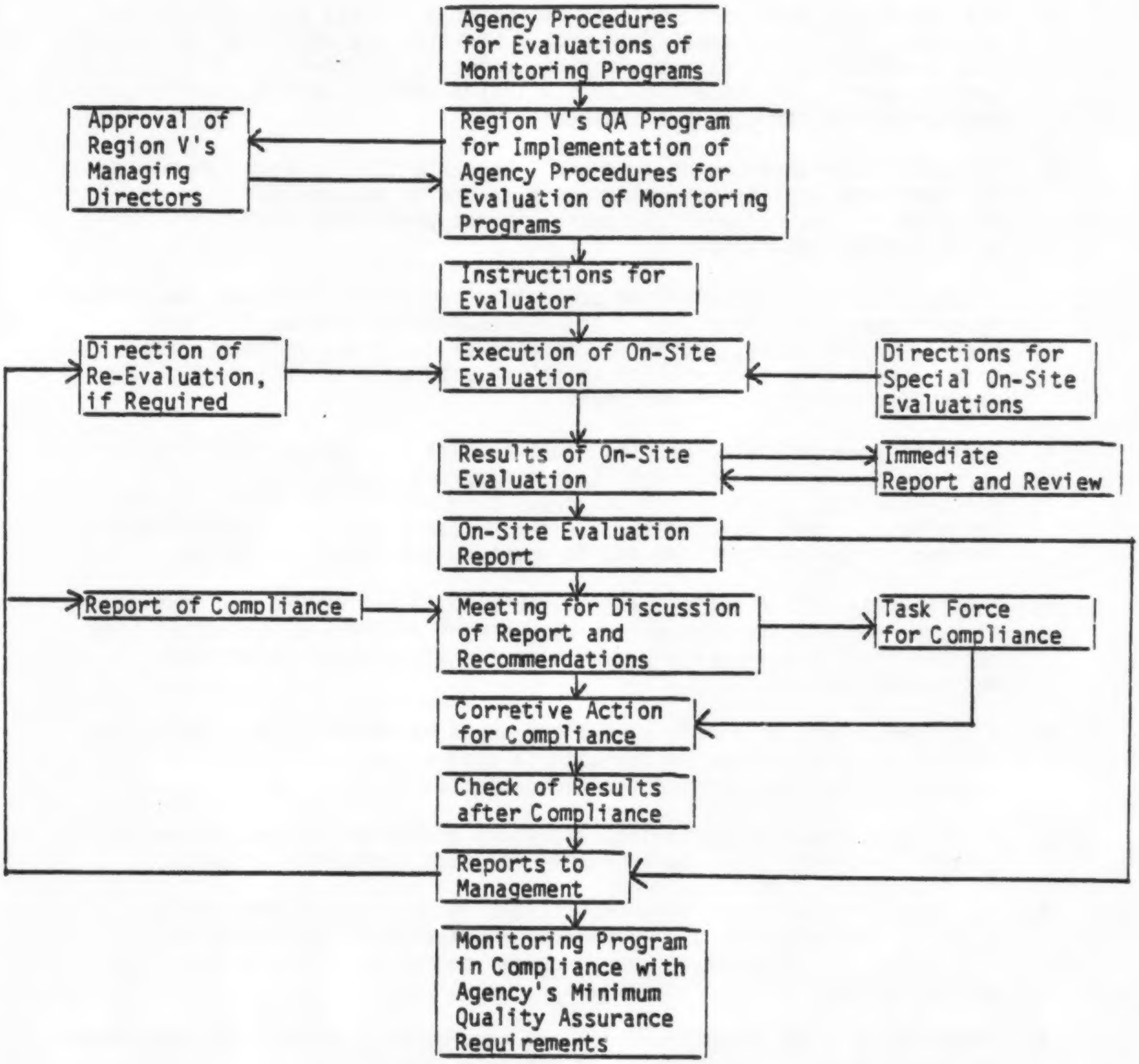
SECTION 4

REFERENCES

1. Eichelberger, J.W., L.E. Harris, and W.L. Budde, "Reference Compound to Calibrate Ion Abundance Measurements In Gas Chromatography - Mass Spectrometry Systems," Anal. Chem., 47, 995 (1975).
2. Budde, W.L., and J.W. Eichelberger, "An EPA Manual for Organics Analysis Using Gas Chromatography - Mass Spectrometry," EPA Report No. EPA 600/8-79-006, March, 1979.
3. Budde, W.L., and J.W. Eichelberger, "Organics Analysis Using GC/MS," Ann Arbor Science Publishers, Ann Arbor, Michigan, July 1979.
4. "Guidelines Establishing Test Procedures for the Analysis of Pollutants," Federal Register
5. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories," EPA Report No. EPA-600/4-79-019, March, 1979, Chapter 6.

APPENDIX 17

LIFE CYCLE OF AN ON-SITE SYSTEM EVALUATION


Agency Procedures for Evaluations of Monitoring Programs
Approval of Region V's Managing Directors
Region V's QA Program for Implementation of Agency Procedures for Evaluation of Monitoring Programs
Instructions for Evaluator
Direction of Re-Evaluation, if Required
Execution of On-Site Evaluation
Directions for Special On-Site Evaluations
Results of On-Site Evaluation
Immediate Report and Review
On-Site Evaluation Report
Report of Compliance
Meeting for Discussion of Report and Recommendations
Task Force for Compliance
Corretive Action for Compliance
Check of Results after Compliance
Reports to Management
Monitoring Program in Compliance with Agency's Minimum Quality Assurance Requirements

APPENDIX 18

ELEMENTS FOR A SECTION 106, 208, 404(b)(1) AND GREAT LAKES NATIONAL PROGRAM MONITORING QUALITY ASSURANCE PROGRAM

1. The laboratory shall document and implement a Quality Assurance Policy to assure sufficient quality control activities are maintained to assure data credibility for each monitoring project. Management or supervisory quality control duties and responsibilities must be defined for its own monitoring and for contract projects.

2. A Quality Assurance Coordinator shall be designated by each laboratory to coordinate quality control activities and to assure that they are being performed. If quality control is not practiced, then there can be no quality assurance.

3. Documented, technique oriented collection procedures shall be implemented by each agency to assure valid and representative samples for surface waters, ground waters, point source discharges, fish, sediment, etc. Uniform record keeping will be established to provide data credibility and sufficient "chain-of-custody".

4. Field measurement methodologies shall be used that are appropriate for each monitoring project. Reference or approved methods must be used for monitoring. Calibration and preventive maintenance protocols are to be established and used for all field instruments and methodologies. Records of the calibrations and maintenance are to be maintained.

5. Sample preservation protocols shall be established by EPA for consistency with the compositing time period used during monitoring, transport time between field and laboratory, and dictates of required laboratory methodologies, etc.

6. A uniform source of sample containers shall be established. Sufficient quality control will be established to assure the appropriateness of containers used for each monitoring project.

7. Sufficient number of field and laboratory personnel trained in quality control practices shall be available for each monitoring project.

8. The laboratory will establish sufficient record keeping and sample handling practices for sample receipt and analyses, consistent with field record keeping practices, in order to maintain data credibility and sufficient "chain-of-custody".

9. Protocols will be established for and records will be kept of instrument calibration and maintenance in an agency's laboratory. Appropriate protocols will be established and used to assure the acceptance of designated laboratory prepared materials (eg. - distilled water) and purchased materials (eg. - microbiology media).

10. Each laboratory will utilize and document methodologies, appropriate in precision, sensitivity and accuracy, for each monitoring project. Reference or approved methods must be used for monitoring and are subject to review by the QAO.

11. Intra-laboratory audits of controls or "spiked" samples, replicate analyses, and reagent blanks are to be utilized, recorded, and documented by each laboratory to assure the acceptance of data for each monitoring project. Summaries or quality control charts for these intra-laboratory audits can be utilized to document analytical performance. The ability of these intra-laboratory aidit data to represent actual data quality is dependent on the specific audits performed and an understanding of their utility by a data user.

12. Inter-laboratory audits of independently prepared reference smples or U.S. EPA quality control samples, when available, are to be used at a minimum frequency of quarterly and their results documented as part of an agency's quality assurance program. Inter-laboratory audits or reference samples assure analytical accuracy and maintenance of calibration accuracy of a laboratory's day-to-day intra-laboratory quality control program.

13. Each laboratory is requested to participate in U.S. EPA's performance sample program, usually scheduled once per year for monitoring agencies. Results should be documented as part of an agency's quality assurance program and can replace one of the above inter-laboratory audits.

14. A quality assurance program should assure that only data meeting acceptance criteria for the above elements are used for each monitoring project. Data in computerized data management or storage systems must be audited or verified as being the same as the actual field and laboratory results.

APPENDIX 19

SUMMARY OF GUIDELINES FOR STATION SITING AND PROBE PLACEMENT*

SITE SELECTION			PROBE PLACEMENT (METERS)		
POLLUTANT	SITE TYPE	SITE LOCATION	HEIGHT ABOVE GROUND	VERTICAL CLEARANCE[a]	HORIZONTAL CLEARANCE[b]
Sulfur dioxide	Peak	Maximum point determined from atmosphere diffusion model, histoical data, emission density, and representative of population exposure.	3 to 15	1 to 2	>2
	Neighborhood	Determined on basis of population patterns and air quality gradients	3 to 15	1 to 2	>2
	Background	Nonurban area within Region	3 to 15	1 to 2	>2
Suspended Particulates	Peak	Same as for SO_2	2 to 15	--	>2
	Neighborhood	Same as for SO_2	2 to 15	--	>2
	Background	Same as for SO_2	2 to 15	--	>2
Ozone	Peak	Representative area downwind of the CBD[c] area 15 to 25 km from downtown and >100 m from major traffic arteries or parking areas	3 to 15	1 to 2	>2
	Neighborhood	Sites in center city, residential, commercial areas	3 to 15	1 to 2	>2
	Background	Nonurban area within Region	3 to 15	1 to 2	>2
Nitrogen dioxide	Peak	Same as for Ozone, except distance of 10 to 15 km	3 to 15	1 to 2	>2
	Neighborhood	Same as for Ozone	3 to 15	1 to 2	>2
	Background	Same as for Ozone	3 to 15	1 to 2	>2

APPENDIX 19 (Continued)

SUMMARY OF GUIDELINES FOR STATION SITING AND PROBE PLACEMENT*

SITE SELECTION			PROBE PLACEMENT (METERS)		
POLLUTANT	SITE TYPE	SITE LOCATION	HEIGHT ABOVE GROUND	VERTICAL CLEARANCE[a]	HORIZONTAL CLEARANCE[b]
Carbon monoxide	Street Canyon	See Supplement A	3 ± 1/2	1	>2
	Neighborhood	See Supplement A	3 ± 1/2	1	>2
	Corrider	See Supplement A	3 ± 1/2	1	>2
	Background	See Supplement A	3 to 10	1	>2
Non-Methane hydrocarbons	Research and Planning	Generally limited to areas of peak emission density of hydrocarbons, i.e., CBD[c]			
Nitric Oxide	Research and Planning	Generally coincident with NMHC sites	3 to 15	1 to 2	>2
Nitrogen Dioxide	Research and Planning	Generally coincident with NHMC sites	3 to 15	1 to 2	>2

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*Guidance for Air Qulity Monitoring Network Design and Instrument Siting (Revised). U.S. Environmental Protection Agency Guideline Series OAQPS No. 1,2-012, September 1975.[1]

[a] Vertical clearance above rooftop or other supporting structure.

[b] Horizontal clearance from side of supporting structure or other restriction to air flow.

[c] Central Business District.

GLOSSARY

Analytical or reagent blank: a blank used as a baseline for the analytical portion of a method. For example, a blank consisting of a sample from a batch of absorbing solution used for normal samples, but processed through the analytical system only, and used to adjust or correct routine analytical results.

Audit: A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which quantitative data are independently obtained for comparison with routinely obtained data, or (2) system audits of a qualitative nature that consist of an on-site review of a laboratory's quality assurance program and physical facilities for sampling, calibration and measurement.

Bioassay: Using living organisms to measure the effect of a substance, factor, or condition.

Biomonitoring: The use of living organisms to test water quality at a discharge site or downstream.

Blank or sample blank: a sample of a carrying agent (gas, liquid, or solid) that is normally used to selectively capture a material of interest and that is subjected to the usual analytical or measurement process to establish a zero baseline or background value, which is used to adjust or correct routine analytical results.

Calculation: The arithmetic conversion of raw analytical data to some standardized dimension form suitable for formating in a data report or for its final intended use. For example, "x" ml/500 ml sample or reagent might be calculated to be 10 mg/liter of zinc chloride which exceeds the discharge limitations of a specific permit.

Calibration: Establishment of a relationship between various calibration standards and the measurements of them by a measurement system (or portions thereof). The levels of calibration standard should bracket the range of levels for which actual measurements are to be made.

Completeness: The amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal operations.

Confidence interval: A value interval that has a designated probability (the confidence coefficient) of including some defined parameter of the population.

Confidence limits: The outer boundaries of a confidence interval.

Contract: The legal instrument reflecting a relationship between the Federal Government and a State or local government or other recipient: (1) whenever the principal purpose of the instrument is the acquisition, by purchase, lease, or barter, of property or services for the direct benefit or use of the Federal Government; or (2) whenever an executive agency determines in a specific instance that the use of a type of procurement contract

Cooperative agreement: The legal instrument reflecting the relationship between the Federal Government and a State or local government or other recipient whenever: (1) the principal purpose of the relationship is the transfer of money, property, services, or anything of value to the State or local government or other recipient to accomplish a public purpose of support or stimulation authorized by Federal statute, rather than acquisition, by purchase, lease, or barter, of property or services for the direct benefit or use of the Federal Government; and (2) substantial involvement is anticipated between the executive agency acting for the Federal Govenrment and the State or local government or other recipient during performance of the contemplated activity.

Data validation: A systematic effort to review data to identify any outliers or errors and thereby cause deletion or flagging of suspect values to assure the validity of the data to the user. This "screening" process may be done by manual and/or computer methods, and it may utilize any consistent technique such as sample limits to screen out impossible values or complicated acceptable relationships of the data with other data.

In-house project: A project carried out by EPA staff in EPA facilities.

Inter-laboratory: Between two different laboratories.

Intra-laboratory: Within a given laboratory.

Measures of dispersion or variability: Measures of the differences, scatter, or variability of values of a set of numbers. Measures of the dispersion or variability are the range, the standard deviation, the variance, and the coefficient of variation.

Performance audit: Planned independent (duplicate) sample checks of actual output made on a random basis to arrive at a quantitative measure of the quality of the output. These independent checks are made by an auditor subsequent to the routine checks by a field technician or laboratory analyst.

Performance test sample: A sample or sample concentrate (to be diluted to a specified volume before analysis) of known (to the EPA only) true value which has been statistically established by interlaboratory tests. These samples are commonly provided to laboratories to test analytical performance. Analytical results are reported to the EPA for evaluation.

Proficiency testing: Special series of planned tests to determine the ability of field technicians or laboratory analysts who normally perform routine analyses. The results may be used for comparison against established criteria, or for relative comparisons among the data from a group of technicians or analysts.

Program: The technical office or staff that has responsibility for a part of the Agency's operation. For R&D grants, the "programs" are the Office of Research and Development, the Office of Air Quality Planning and Standards, the Office of Solid Waste Management Programs, and the Office of Mobile Sources Air Pollution Control.

Project officer: The EPA official designated in the grant or contract agreement as the Agency's principal contact with the grantee on a particular grant. This person is the individual responsible for project monitoring and for recommendations on or approval of proposed project changes.

Quality: The totality of feature and characteristics of a product or service that bears on its ability to satisfy a given purpose. For pollution measurement systems, the product is pollution measurement data, and the characteristics of major importance are accuracy, precision, and completeness. For monitoring systesm, "completeness", or the amount of valid measurements obtained relative to the amount expected to have been obtained, is usually a very important measure of quality. The relative importance of accuracy, precision, and completeness depends upon particular purpose of the user.

Quality Assurance: (1) An organization's total program for assuring the reliability of the data it produces.

(2) A system for integrating the quality planning, quality assessment, and quality improvement efforts of various groups in an organization to enable operations to meet user requirements

at an economical level. In pollution measurement systems, quality assurance is concerned with all of the activities that have an important effect on the quality of the pollution measurements, as well as the establishment of methods and techniques to measure the quality of the pollution measurements. The more authoritative usages differentiate between "quality assurance" and "quality control", where quality control is "the system of activities to provide a quality product" and quality assurance is "the system of activities to provide assurance that the quality control system is performing adequately".

Quality assurance manual: An orderly assembly of management policies, objectives, principles, and general procedures by which an agency or laboratory outlines how it intends to produce quality data.

Quality assurance plan: An orderly assembly of detailed and specific procedures by which an agency or laboratory delineates how is produces quality data for a specific project or measurement method. A given agency or laboratory would have only one quality assurance manual, but would have a quality assurance plan for each of its projects or programs (group of projects using the same measurement methods; for example, a laboratory service group might develop a plan by analytical instrument since the service is provided to a number of projects).

Quality control: The detailed and specific procedures used to insure the quality of data produced by a particular measurement activity; the system of activities designed and implemented to provide a quality product.

Quality control (internal): The routine activities and checks, such as periodic calibrations, duplicate analyses, use of spiked sample, etc., included in normal internal procedures to control the accuracy and precision of a measurement process.

Quality control (external): The activities which are performed on an occasional basis, usually initiated and performed by persons outside normal routine operations, such as on-site system surveys, independent performance audits, interlaboratory comparisons, etc., to assess the capability and performance of a measurement process.

Range: The difference between the maximum and minimum values of a set of values. When the number of values is small (i.e., 12 or less), the range is a relatively sensitive (efficient) measure of variability.

Reagent: A chemical material, usually a compound of high purity, which is used as a reactant in the process of a chemical analysis.

Recovery: That percentage of a parameter in a sample which is detected or "recovered" from that sample during chemical analysis.

Reliability: A numerical statement of accuracy and precision.

Representativeness: A numerical statement of how well a sample or group of samples or the data derived therefrom represents the actual parameter variations at the sampling point, plus how well that sampling point represents the actual parameter variations which are under study.

Sample: A subset or group of objects or things selected from a larger set called the "lot" or "population". The objects or things may be physical, such as specimens for testing, or they may be data values representing physical samples. Unless otherwise specified, all samples are assumed to be randomly selected. Samples can take numerous forms, such as:

Representative sample: A sample taken to represent a lot or population as accurately and precisely as possible. A representative sample may be either a completely random sample or a stratified sample, depending upon the objective of the sampling and the conceptual population for a given situation.

Spiked sample: A normal sample of material (gas, solid, or liquid) to which is added a known amount of some substance of interest. The extent of the spiking is unknown to those analyzing the sample. Spiked samples are used to check on the performance of a routine analysis or the recovery efficiency of a method.

Standard deviation: The square root of the variance of a set of values:

$$s = \frac{{}^{n}_{i=1}\,(X_i - X)^2}{n-1}$$

if the values represent a sample from a larter population:

$$\frac{{}^{N}_{i=1}\,(X_i - u)^2}{N}$$

where u is the true arithmetic mean of the population. The property of the standard deviation that makes it most particularly meaningful is that it is in the same units as the values of the set, and universal statistical tables for the normal (and other) distributions are expressed as a function of the standard deviation. Mathematically, the tables could just as easily be expressed as a function of the variance.

Standard reference material (SRM): A material produced in quantity, of which certain properties have been certified by the National Bureau of Standards (NBS) or other agencies to the extent possible to satisfy its intended use. The material should be in a matrix similar to actual samples to be measured by a measurement system or be used directly in preparing such a matrix. Intended uses include: (1) standardization of solutions, (2) calibration of equipment, and (3) monitoring the accuracy and precision of measurement systems.

Standard reference sample: A carefully prepared material produced from or compared aganist an SRM (or other equally well characterized material) such that there is little loss of accuracy. The sample should have a matrix similar to actual samples used in the measurement system. These samples are intended for use primarily as reference standards to: (1) determine the precision and accuracy of measurement systems, (2) evaluate calibration standards, and (3) evaluate quality control reference samples. They may be used "as is" or as a component of a calibration or quality control measurement system. Examples: an NMS-certified sulfur dioxide permeation device is an SRM. When used in conjunction with an air dilution device, the resulting gas becomes an SRS. An NBS-certified oxide gas is an SRM. When diluted with air, the resulting gas is an SRS.

Standardization: A physical or mathematical adjustment or correction of a measurement system to make the measurements conform to predetermined values. The adjustments or corrections are usually based on a single-point calibration level.

Calibration standard: A standard prepared by the analyst for the purpose of calibrating an instrument. Laboratory control standards are prepared independently from calibration standards for most methods.

Detection limit: That number obtained by adding two standard deviations to the average value obtained for a series of reagent blanks that are analysed over a long time period (several weeks or months).

Duplication analyses: The collection of two samples from the same field-site which are analyzed at different times but usually on the same day.

Laboratory control standard: A standard of known concentration prepared by the analyst.

Reference standard: A solution obtained from an outside source having a known value and analyzed as a blind sample.

Relative percent error for duplicate analyses: The difference between the measured concentration for the duplicate pair times 100 and divided by the average of the concentration.

Relative percent error for laboratory control standards: The difference between the measured value and the theoretically correct value times 100 and divided by the correct value.

Relative percent error of a reference sample analysis: The difference between the correct and measured values times 100 and divided by the correct concentration.

Standards based upon usuage:

Calibration standard: A standard used to quantitate the relationship between the output of a sensor and a property to be measured. Calibration standards should be traceable to standard reference materials or primary standard.

Quality control reference sample (or working standard): A material used to assess the performance of a measurement or portions thereof. It is intended primarily for routine intralaboratory use in maintaining control of accuracy and would be prepared from or traceable to a calibration standard.

Standards depending upon "purity" or established physical or chemical constants:

Primary standard: A material having a known property that is stable, that can be accurately measured or derived from established physical or chemical constants, and that is readily reproducible.

Secondary standard: A material having a property that is calibrated against a primary standard.

Standards in naturally-occurring matrix: Standards relating to the pollutant measurement portions of air pollution measurement systems may be categorized according to matrix, purity, or use. Standards in a naturally-occurring matrix include Standard Reference Materials and Standard Reference Samples.

Statistical control chart (also Shewhart control chart): A graphical chart with statistical control limits and plotted values (usually in chronological order) of some measured parameter for a series of samples. Use of the charts provides a visual display of the pattern of the data, enabling the early detection of time trends and shifts in level. For maximum usefulness in control, such charts should be plotted in a timely manner, i.e., as soon as the data are available.

System audit: A systematic on-site qualitative review of facilities, equipment, training, procedures, record-keeping, validation, and reporting aspects of total (quality assurance) system to arrive at a measure of the capability and ability of the system. Even though each element of the system audit is qualitative in nature, the evaluation of each element and the total may be quantified and scored on some subjective basis.

Systematic error: The condition of a consistent deviation of the results of a measurement process from the reference or known level.

Test Variability: Accuracy: The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100(X-T)/T, and sometimes expressed as a ratio, X/T.

Bias: A systematic (consistent) error in test results. Bias can exist between test results and the true value (absolute bias, or lack of accuracy), or between results from different sources (relative bias). For example, if different laboratories analyze a homogeneous and stable blind sample, the relative biases among the laboratories would be measured by the differences existing among the results from the different laboratories. However, if the true value of the blind sample were known, the absolute bias or lack of accuracy from the true value would be known for each laboratory.

Precision: A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is most desirably expressed in terms of the standard deviation but can be expressed in terms of the variance, range, or other statistics. Various measures of precision exist depending upon the "prescribed similar conditions".

Replicates: Repeated but independent determinations of the same sample, by the same analyst, at essentially the same time and same conditions. Care should be exercised in considering replicates of a portion of an analysis and replicates of a complete analysis. For example, duplicate titrations of the same digestion are not valid replicate analyses, although they may be valid replicate titrations. Replicates may be performed to any degree, e.g., duplicates, triplicates, etc.

Reproducibility: The precision, usually expressed as a standard deviation, measuring the variability among results of measurements of the same sample at different laboratories.

Validation: A systematic effort to review data to identify outliers or errors and thereby cause deletion or flagging of suspect values to assure the validity of the user's data.

Variance: Mathematically, for a sample, the sum of squares of the differences between the individual values of a set and the arithmetic mean of the set, divided by one less than the number of values.

Verification: Follows validation and permits the certification of the data for an intended legal use, presuming that the chain-of-custody requirements are found to be intact. Again the terminology used in this document is intended to be general and should in no way be construed to limit the use of special area terminologies in the preparation of the required QA Plan.

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